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Original Paper

Montmorillonite modified by composite modifier as a rheological regulator of drilling fluid suitable for ultra-low temperature conditions in Antarctica

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A B S T R A C T

In order to settle the issues of poor rheology for drilling fluids in Antarctica, it is important to develop an agent that can availably address these challenges. For this reason, a rheological regulator (HSCN) of drilling fluid was synthesized by modifying montmorillonite with composite modifiers (DODMAC and CPL). The structure of HSCN was characterized by X-ray diffraction, contact angle, infrared spectroscopy and scanning electron microscopy. And HSCN properties were also evaluated by experiments such as colloidal rate, rheology, viscosity-temperature characteristics and corrosion test. Finally, the mechanism of HSCN was investigated. 2% HSCN can enhance the improvement rate of yield point for drilling fluid at −55 °C by 167%, and the colloidal rate of drilling fluid is 90.4% after 24 h. The corrosion of the three rubbers is weak, with a maximum mass increase of only 0.014 g and a maximum outside diameter increase of 0.04 cm. The mechanism study shows that the staggered lapping between HSCN lamellar units forms an infinitely extended reticular structure. The structure is mainly formed by the electrostatic attraction between HSCN particles, hydrogen bonding, physical adsorption and entanglement between the long carbon chains in HSCN. The formation of this structure can effectively enhance the rheology properties of drilling fluids. This research gives a direction for the investigation of drilling fluids suitable for Antarctic conditions, which is greatly sense for accelerating the efficient exploitation of oil and gas in Antarctica.

1. Introduction

As the increasing exploitation of oil and gas resources, polar regions have been regarded as one of the important targets for exploitation by many countries. Antarctica has rich resources, and more than 220 kinds of minerals have been found, mainly including oil, natural gas, iron, coal, copper, diamond and so on. At present, the oil reserves in Antarctica are about (7.95–15.90) × 10^9 m^3, and the natural gas reserves are approximately (3–5) × 10^{12} m^3. However, due to extremely low surface temperature and complicated geological environment (Rohling et al., 2009; Turner et al., 2020) in Antarctica, it poses significant challenges for the performances of drilling fluids. The distribution of ice sheet temperature with depth in different regions of the Antarctic is shown in Fig. 1 (Mellor, 1960; Talalay et al., 2020).

Drilling fluid is an important component of drilling engineering, playing a crucial role in carrying rock cuttings, balancing formation pressure, obtaining formation information and assisting in rock breaking during the drilling process. It is one of the key technologies for polar oil and gas drilling. Antarctic drilling includes snow drilling, ice drilling and sub ice rock drilling (Talalay et al., 2016). Currently, the commonly used polar drilling fluids include four types (Gosink et al., 1994; Bentley and Koci, 2007; Talalay et al., 2011), namely petroleum drilling fluids (Morgan et al., 1994; Mulvaney et al., 2007), alcohol drilling fluids (Gosink et al., 1993; Talalay et al., 2019), ester drilling fluids (Gosink et al., 1991; Fujita et al., 1994; Grootes et al., 1994; Talalay et al., 2014; Motoyama et al., 2021) and silicone oil-based drilling fluids (Talalay, 2007).

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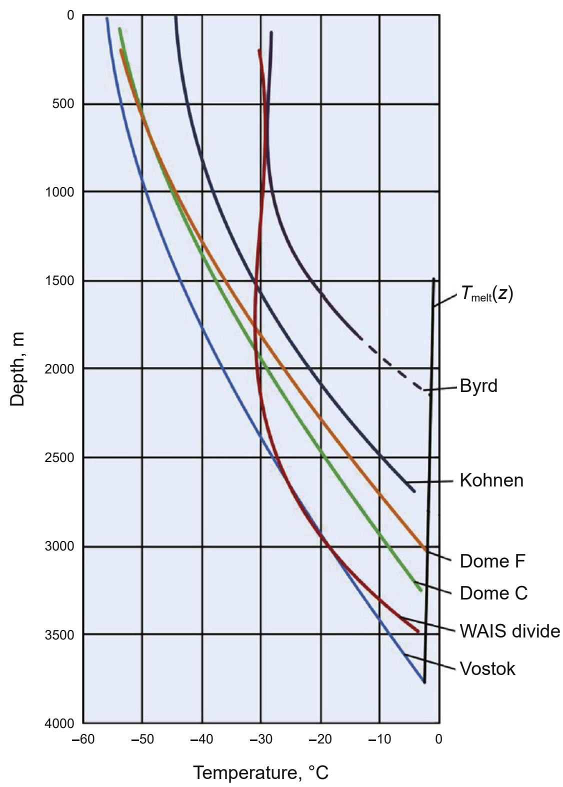

Fig. 1. Variation diagram of ice sheet temperature with depth (Talalay et al., 2020).

The drilling of ice cores in European Antarctica was carried out using de-aromatised solvent oil (Exxsol D30) as drilling fluid. During the drilling process, the accumulation of ice debris in the wellbore led to low drilling efficiency and even difficulties in drilling (Augustin et al., 2007). However, as research continues to deepen, it was found that ethylene glycol drilling fluid had a good ability to prevent the adhesion of ice debris in ice formations above −30 °C (Talalay et al., 2015). However, at lower temperatures, this type of drilling fluid is no longer suitable, and it can affect the stability of the wellbore (Johnsen et al., 2007). Ester drilling fluids were also used for Antarctic ice core drilling. ESTISOL™ ester drilling fluid was once used as the SPICE core project to obtain high-quality ice cores of 1751 m at an Antarctic drilling site with an average annual temperature of −51 °C (Johnson et al., 2021). However, it caused physical discomfort to the researchers and reacted with sealing components and steel wire sheath materials. Butyl acetate exhibits good performance at low temperatures (Zhang et al., 2014), and it was used to achieve a drilling depth of 800 m in Dome A area of southeastern Antarctica (Hu et al., 2021). However, butyl acetate poses a great threat to human health and has strong corrosiveness. Overall, the drilling fluids used in the Antarctic region mentioned above mainly suffer from insufficient low temperature resistance, strong corrosiveness, high toxicity and poor rheological properties. However, the poor rheological performance is mainly manifested by problems such as low yield point and poor ability to suspend ice debris. The above issues may make poor carrying abilities of ice debris for drilling fluids, leading to the accumulation of ice debris at the bottom of the well and abnormalities of drilling parameters, resulting in downhole accidents. Inevitably, the above issues will lead to an increase in drilling costs.

At present, diesel and aviation kerosene (Vasiliev and Talalay, 2011; Shturmakov et al., 2014; Kuhl., 2021) are the main working fluids for Antarctic drilling. However, diesel is rarely used in Antarctic drilling due to its not low freezing point, high irritancy, toxicity and corrosiveness (Sun et al., 2022). Compared with water-based drilling fluids, it has excellent low-temperature resistance, lubricity, inhibition. However, oil-based drilling fluids still have poor rheology properties at ultra-low temperature. Therefore, it is of great significance to develop an additive that can effectively improve the rheology performances at ultra-low temperature. In the research of rheological regulators for high-temperature drilling fluids, experts and scholars have developed organoclays (Power and Zamora, 2003; Li et al., 2014), fatty acid amides (Huang et al., 2016), oil soluble polymers (Basfar et al., 2020) and nano-composites (Madkour et al., 2016). It is believed that organic montmorillonite has great potential in enhancing the rheology performances of oil-based drilling fluids, and found that a large crystalline layer spacing, good lipophilicity and strippability are important factors affecting the rheology performances of drilling fluids (Zhuang et al., 2019a, b, c). Shi et al. synthesized a rheological regulator belonged to dendritic polymer using dodecyl dicarboxylic acid and triethylenetetramine as raw materials, which can improve the yield point from 0.27 to 1.18 Pa (Shi et al., 2018). Noah et al. prepared a polymer nanocomposites using zinc oxide, modified calcium carbonate and polystyrene butadiene rubber copolymer as raw materials (Noah et al., 2017), and the rheological regulator can increase the yield point of 6 Pa at 150 °C. Yılmaz et al. developed a rheological regulator based on flaxseed oil, glycerol, diisocyanate and alkyd resin as raw materials (Yılmaz et al., 2005). It has an excellent performance that the viscosity ratio of low shear rate (10 s^{-1}) and high shear rate (100 s^{-1}) of drilling fluid can be increased to 1.191, which shows good cuttings-carrying and suspension performance. The above related studies only focused on the development of rheological regulators suitable for high-temperature formations. However, the research field of drilling in Antarctica is basically a major blank, and there have been no reports on the study of rheological regulators for ultra-low temperature drilling fluids in Antarctica. Therefore, it is urgent to develop ultra-low temperature rheological regulators suitable for the Antarctic region, so as to enhance the suspension and carrying properties of drilling fluid on ice debris and rock cuttings, and thus enhance the drilling efficiency.

In this paper, a new type of rheological regulator (HSCN) of drilling fluids for the Antarctic region was prepared by modifying montmorillonite with composite modifiers (DODMAC and CPL). The synthesized product was structurally characterized by infrared spectral scanning, contact angle test, scanning electron microscopy and XRD test. The colloidal rates, rheological properties, viscosity-temperature characteristics and corrosiveness at ultra-low temperatures were evaluated. Finally, the mechanism of the rheological regulator in drilling fluid was investigated. It is of much importance in guiding the further advancement of Antarctic scientific research. The work chart of this study is shown in Fig. 2.

2. Materials and methods

2.1. Materials

Montmorillonite (AR) was supplied by Anhui Zesheng Technology Co., Ltd; Dimethyl dioctadecyl ammonium chloride (AR) and caprolactam (AR) were from Aladdin Chemical Reagent; Hanke wetting agent (DNS-18 models) was supplied by Hanke Chemical Technology Co., Ltd; Number 4 aviation kerosene was supplied by Jinan Xiangtai Chemical Co., Ltd; Number 5 white oil (QY models) was supplied by Shandong Youwei Chemical Co., Ltd.

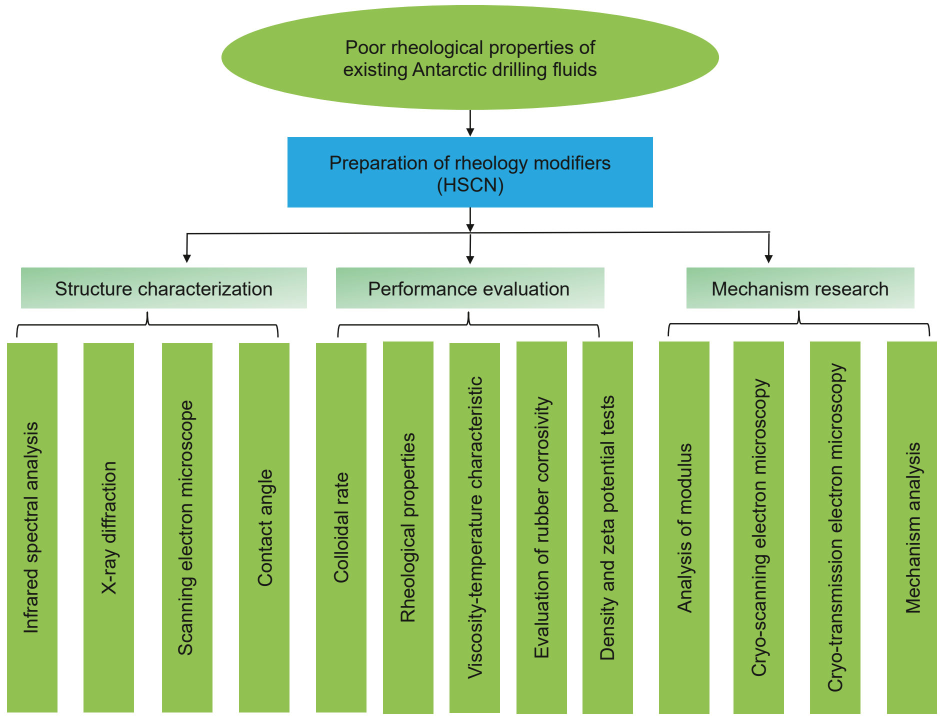

Fig. 2. Work chart showing all processes in the research works.

2.2. Preparation of rheological regulator

Montmorillonite suspension was made up by weighing 10 g montmorillonite and dissolving it in 115 g water, and stirred at 25 °C for 24 h to hydrate it. Afterwards, 2.81 g of dimethyl dioctadecyl ammonium chloride (DODMAC) and 0.94 g of caprolactam (CPL) were weighed, with a total concentration of 2 wt%, and dissolved in 184 g water to prepare a modified liquid and stirred for 10 min. Subsequently, the montmorillonite suspension was heated up to 80 °C during stirring, and the modified liquid was gradually poured into it. The reaction was maintained at 80 °C for 1.5 h. It was centrifuged by TG16-WS centrifuge (Changsha Xiangyi Technology Co., Ltd) at 8000 rpm for 10 min when the reaction was finished. Finally, the rheological regulator (HSCN) was obtained by drying at 80 °C to constant weight, crushing with a pulverizer and passing through a 200 mesh sieve. The structure of the rheology regulator is shown in Fig. 3.

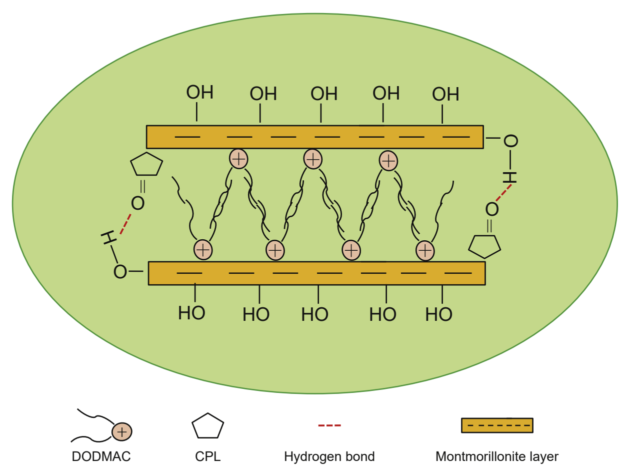

Fig. 3. Structure diagram of rheological regulator (HSCN).

2.3. Structural characterization of HSCN

2.3.1. Infrared spectral analysis

Infrared analysis was carried out using a Fourier Transform Infrared Spectroscopy scanner (PE Co., Ltd., USA), and the samples were analyzed by a method of KBr compression with a resolution ratio of 4 cm^{-1}.

2.3.2. Analysis of X-ray diffraction

Using X-ray diffractometer model D/MAX-C (Rika Co., Ltd, Japan) to measure specimen layer spacing. The main parameters include the use of Cu targets, a tube voltage of 40 KV, and a tube current of 25 mA.

2.3.3. Analysis of scanning electron microscopy

Using scanning electron microscope model 450 (FEI Technology Co., Ltd, USA) to characterize the micro-morphology of montmorillonite and rheological regulator (HSCN). Before observation, the specimen powder was coated on a silicon wafer, and gold was sprayed on its surface.

2.3.4. Contact angle analysis

The lipophilicity of the rheological regulators and montmorillonite was analyzed by a contact angle meter manufactured by Dataphysics, Germany. The measurement range was 0°–180°, and the samples were prepared on a YPJ-40T tablet press and tested separately using the droplet method with 5.0 μL of oil droplets as a probe with the same weight. At least five points in different areas were tested, and the contact angle was the average of five points.

2.4. Preparation of base fluid for Antarctica drilling fluid

Number 4 aviation kerosene and Number 5 white oil were prepared into a base oil at a ratio of 7:3, with a total volume of 400 mL, then adding 3% Hanke wetting agent to the base oil. Subsequently, it was stirred for 20 min in a FJ200-SH homogenizer to make a base fluid for Antarctica drilling fluid (Huang et al., 2023).

2.5. Evaluation of HSCN performance

2.5.1. Colloid rate test

A specific concentration of rheological regulator (HSCN) was poured into the base fluid and stirred for 15 min using a high-speed stirrer to mix it thoroughly. Subsequently, 25 mL of the prepared drilling fluid was poured into a stoppered colorimetric tube and left to stand for 90 min, 16 h, and 24 h, respectively, in a low-temperature thermostat to measure the colloid rate. The colloid rate was calculated by the formula (Zhuang et al., 2017; Geng et al., 2019):

$$Cr = \frac{V}{100.0} \times 100\% \tag{1}$$

where Cr - colloid rate, percent; V - colloidal volume, mL.

2.5.2. Rheology test

In accordance with American Petroleum Institute (API) standards (Institute, 2009), using the ZNN-D6 viscometer of ultra-low-temperature (Qingdao Haitongda Technology Co., Ltd.) to measure the variations of rheology parameters at different temperatures, as well as the effect of HSCN dosages on the rheological properties at −55 °C. The calculation formula for rheology parameters is (Zhou and Pu, 2022):

$$AV = 0.5 \times \Phi_{600} \tag{2}$$

$$PV = \Phi_{600} - \Phi_{300} \tag{3}$$

$$YP = 0.5 \times \Phi_{600} - PV \tag{4}$$

where AV–apparent viscosity, mPa·s; PV–Plastic viscosity, mPa·s; YP—yield point, Pa.

The gel strength describes the suspension performance of drilling fluid under static conditions, which is respectively the gel strength of drilling fluid for 10 s and 10 min (Soares et al., 2017).

The base fluid, drilling fluid with 2% and 4% HSCN were frozen (−55 °C) in a thermostat (Jintan Co., Ltd., Changzhou, China) for 16 h, respectively. Subsequently, the variation of drilling fluid viscosity with shear rate was studied by Haake MARS rheometer.

2.5.3. Evaluation of corrosiveness to rubber

Three different materials of seals (silicone rubber, fluoroelastomer, nitrile rubber) were selected and put into base fluid and the drilling fluid with 2% HSCN to freeze for a specific period of time under the condition of −55 °C, and the changes of the outer diameter and mass of the rubber of different materials over time were tested at the end of the freezing to investigate the corrosiveness of HSCN to the seals of different materials (Liu et al., 2016).

2.5.4. Density and zeta potential tests

The rationality of drilling fluid density is an important guarantee for drilling safety. The densities of the base fluid and drilling fluid with 2% HSCN were tested at different temperatures using the SH112EM low-temperature density measuring instrument (Shandong Shengtai Instrument Co., Ltd., China).

The stability of drilling fluid colloid can be analyzed by Zeta potential (Murtaza et al., 2021). Firstly, the base fluid and drilling fluid with 2% HSCN were frozen at −55 °C for 16 h, and then the zeta potential of the samples was measured by a Zetasizer Nano ZS90 (Malvern Instruments Ltd, UK).

2.6. Mechanism research

2.6.1. Analysis of drilling fluid modulus

The base fluid, and drilling fluid with 2% HSCN were frozen (−55 °C) in an ultra-low temperature chamber (Cangzhou Xinxing Co., Ltd., China) for 16 h, respectively. And then the Haake MARS rheometer was operated to study the variation of the drilling fluid modulus under different shear stresses (Werner et al., 2017).

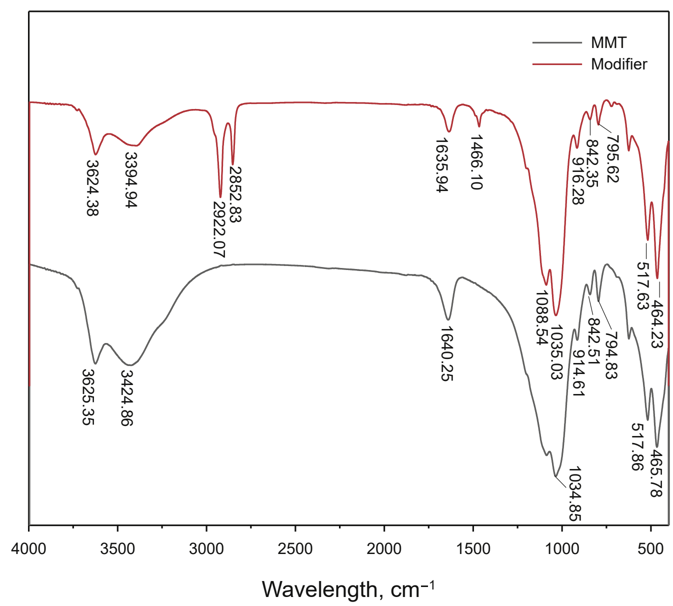

Fig. 4. Infrared spectra of MMT and rheological regulator.

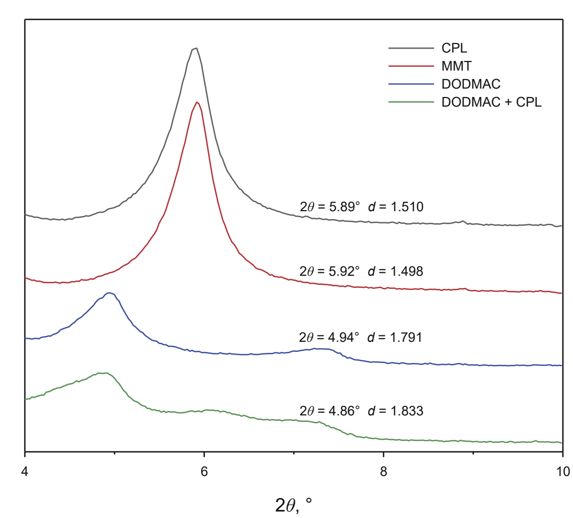

Fig. 5. XRD images of MMT and rheological regulators.

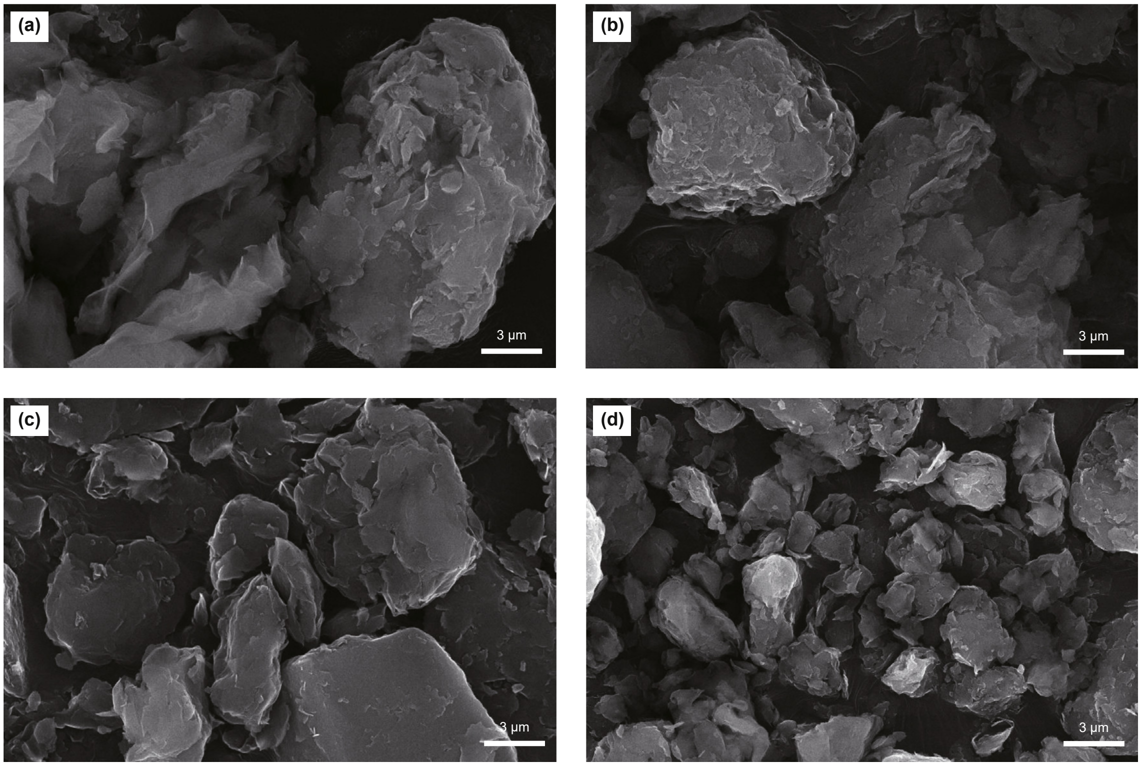

Fig. 6. SEM images of MMT and rheological regulators ((**a**) MMT; (**b**) CPL-MMT; (**c**) DODMAC-MMT; (**d**) (CPL + DODMAC)-MMT).

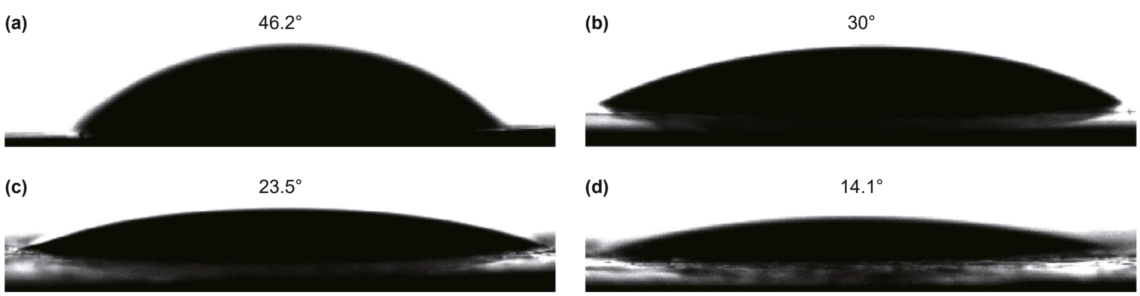

Fig. 7. Contact angle diagram ((**a**) MMT; (**b**) CPL-MMT; (**c**) DODMAC-MMT; (**d**) (CPL + DODMAC)-MMT).

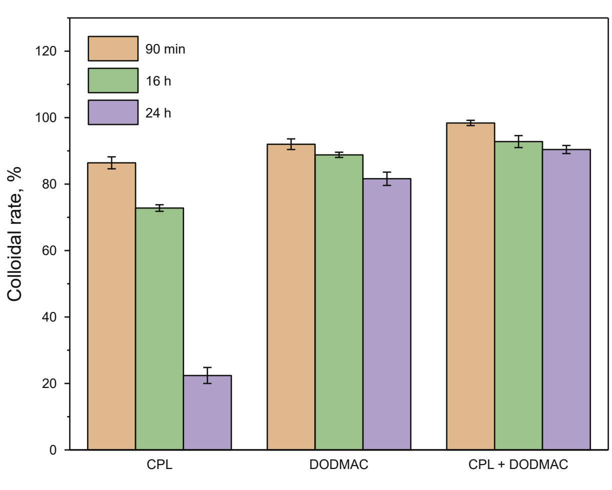

Fig. 8. Effect of different modifiers on the colloidal rate of rheological regulators ((**a**) CPL-MMT; (**b**) DODMAC-MMT; (**c**) (CPL + DODMAC)-MMT).

2.6.2. *Analysis of cryo-scanning electron microscopy*

Cryo-SEM was operated to observe the surface morphology of drilling fluids. The samples were added to the holder and quickly immersed in ultra-low temperature liquid nitrogen (−160 °C). After 2 min, the samples were transferred to a high vacuum freezing environment, and the frozen fracture of the samples leaked out of the fresh section and gold sprayed (Huang et al., 2018). Finally, the samples were frozen, and the cross section of the samples was investigated at ultra-low temperature (−160 °C) to analyze the changes in microscopic morphology.

2.6.3. *Analysis of cryo-transmission electron microscopy*

The microstructure inside the drilling fluid was observed by cryo-transmission electron microscopy. The drilling fluid was first ultrasonically dispersed for 30 min, and then a small drop of the sample was placed on an ultrathin carbon film and dried. The microscopic morphology of drilling fluid was observed by high-resolution cryo-TEM (Segad et al., 2012).

3. Results and discussion

3.1. Structural characterization of HSCN

3.1.1. Infrared spectral analysis

The infrared spectra of montmorillonite and rheological regulator are shown in Fig. 4. In the montmorillonite curve, 3625 cm^{-1} is the stretching vibration peak of the −OH bond, and 3424 cm^{-1} is the stretching vibration peak of the H−O−H bond of the intergranular layer water, corresponding to the H−O−H bond near 1640 cm^{-1}. There is a strong peak of the Si−O−Si bond near 1034 cm^{-1}. In the rheological regulator curve, 3624 cm^{-1} is the peak of −OH bond expansion and vibration; 3394 cm^{-1} is the absorption peak of −NH bond; 2922 cm^{-1} is the absorption peak of −C−H bond; The absorption peak 2852 cm^{-1} is the symmetric expansion and vibration of $-CH_2$; 1466 cm^{-1} is the absorption peaks of $-CH_3$ and $-CH_2$, and 1635 cm^{-1} is the result of the −C=O in the amide interacting with the −OH in the montmorillonite to form hydrogen bonds, which shifts the frequency of the −C=O stretching vibration in the low-wave direction.

3.1.2. X-ray diffraction

Rheological regulator is prepared by organic reaction of montmorillonite and organic modifiers. Since the cation exchange reaction in the process of organic modification will increase the distance between the montmorillonite layers, the results of organic modification are judged by X-ray diffraction (XRD).

Fig. 5 indicates the XRD spectra of montmorillonite and the rheological regulators prepared through different modifiers. The diffraction peak of montmorillonite appeared at 2θ of 5.92°, which corresponds to a layer spacing of 1.498 nm. When montmorillonite was modified by caprolactam (CPL), the diffraction peak 2θ was 5.89°, which corresponds to a layer spacing of 1.510 nm. This indicates that the layer spacing of montmorillonite was almost unchanged after it was modified by CPL. This is because when modified with CPL alone, CPL cannot form hydrogen bonds in a hydrophilic environment and therefore does not increase the layer spacing of montmorillonite. However, the diffraction peak 2θ of DODMAC-modified montmorillonite appears at 4.94°, which corresponds to a layer spacing of 1.791 nm, indicating that the double-chained alkyl quaternary ammonium salts are successfully inserted into the montmorillonite crystal layer, which in turn increases the layer spacing. In addition, the diffraction peak 2θ of montmorillonite modified by composite modifier (DODMAC + CPL) is 4.86°, which corresponds to a layer spacing of 1.833 nm, indicating that the layer spacing of the rheological regulator (HSCN) modified by composite modifier is the largest. This is because the cations in the organic modifier successfully entered the montmorillonite interlayer replacing the inorganic cations in the interlayer, and the fatty chains attached to the quaternary ammonium salt cations are inserted into the interlayer. The occurrence of this ion-exchange reaction resulted in the increased lipophilicity of the clay mineral (Wu et al., 2015), which can be better dispersed in organic solvents. In addition, the carbonyl group in caprolactam and hydroxyl group in the edge of montmorillonite layer form hydrogen bonds in the oil-wet environment, which further increases the layer spacing.

3.1.3. Scanning electron microscope

Scanning electron microscopy (SEM) of montmorillonite and rheological regulators prepared through different modifiers is revealed in Fig. 6.

The SEM graph of montmorillonite indicates that it is composed of a large aggregate of particles, and the particles exist in a clustered lamellar form. The SEM image of the rheological regulator prepared by CPL is similar to the sample of montmorillonite, showing large granular aggregate and close packing between particles. The particles of the rheological regulator modified with DODMAC were significantly smaller in comparison, but large particles were still visible and the particles were closely packed with lamellar montmorillonite. The particles of the rheological regulator (HSCN) modified by DODMAC and CPL contained more stripped clay layers, and the particles were significantly smaller (Fig. 6(d)). This is mainly due to the modifier of double-chain quaternary ammonium increases the spacing between the montmorillonite crystal layers. At the same time, hydrogen bonding is formed by caprolactam and montmorillonite to further increase the spacing between the crystal layers, so that the modifier interpolates the montmorillonite more sufficiently, resulting in a significant reduction of the HSCN particles, and the particles are arranged in a loose manner (Zhuang et al., 2019a, b, c). This existence form enhances the dispersion of the rheological regulator, improving the rheology properties of the drilling fluid.

3.1.4. Contact angle

The dispersion properties of rheological regulator directly influence the rheology performances. The lipophilicity of the

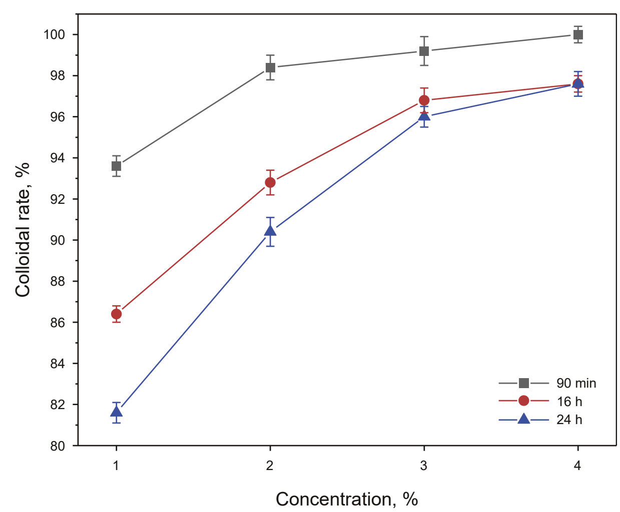

Fig. 9. Variation of colloid rate with HSCN concentration.

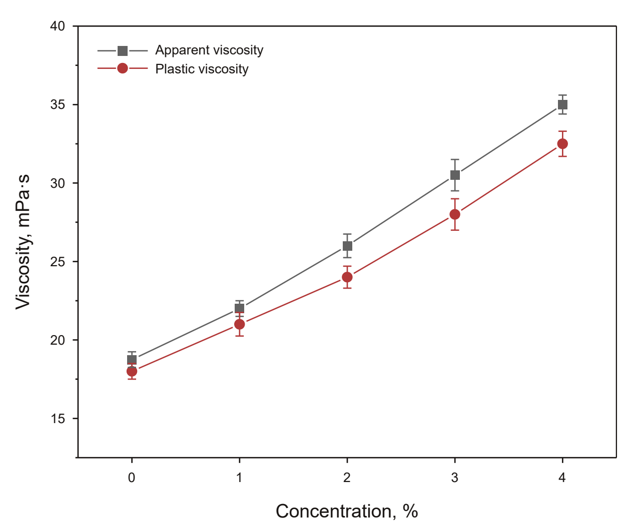

Fig. 10. Variation of drilling fluid viscosity with HSCN concentration.

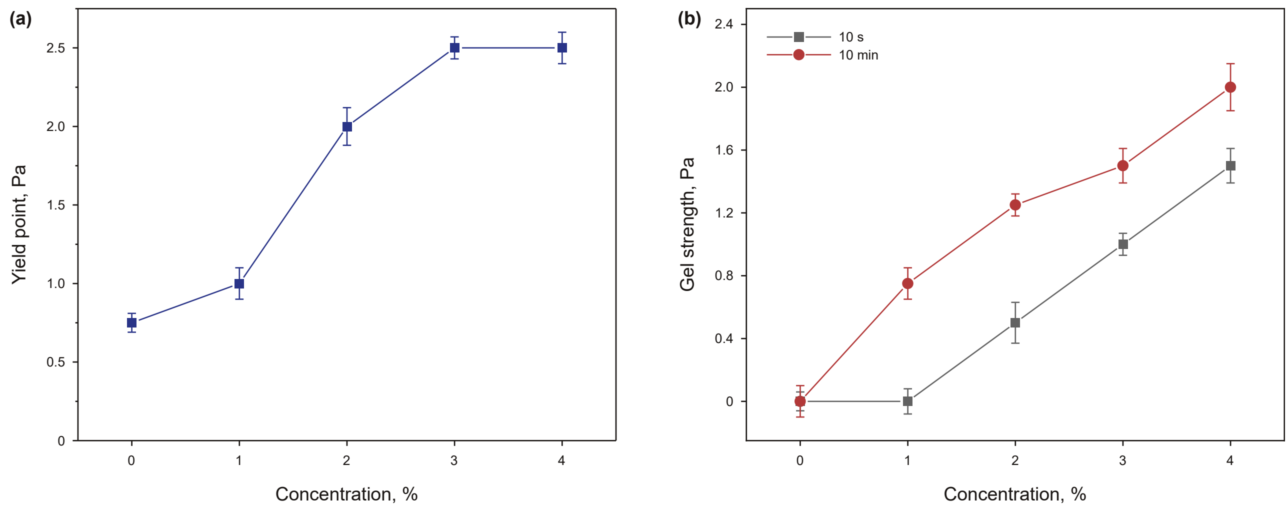

Fig. 11. Variation of yield point and gel strength of drilling fluid with HSCN concentration ((**a**) yield point; (**b**) gel strength).

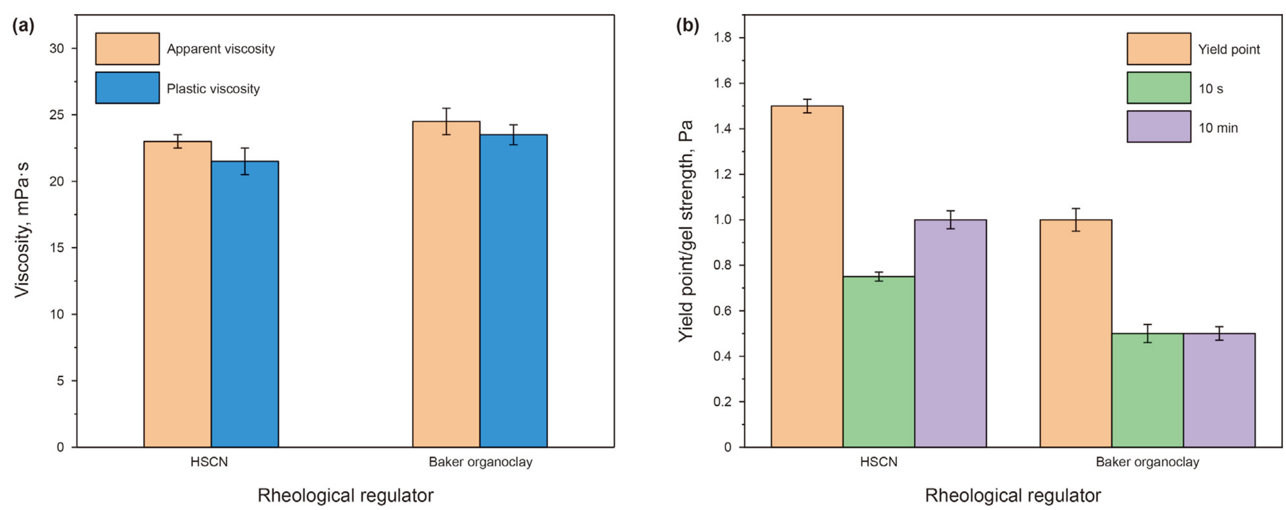

Fig. 12. Comparison of rheological properties between HSCN and Baker organoclay ((**a**) viscosity; (**b**) yield point and gel strength).

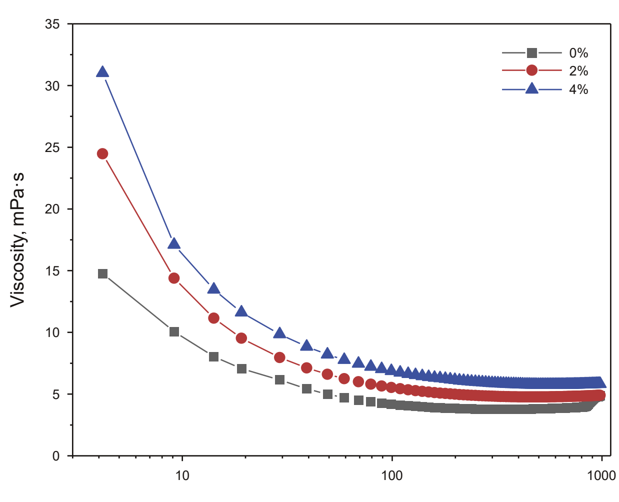

Fig. 13. Variation of drilling fluid viscosity with shear rate.

rheology regulator directly determines its dispersion in drilling fluid. The contact angles of the montmorillonite and rheological regulators (Fig. 7) reflect the difference in lipophilic capacities.

The contact angle of montmorillonite is 46.2°, and the contact angles of the rheological regulators prepared by CPL, DODMAC and CPL + DODMAC are 30°, 23.5° and 14.1°, respectively (Fig. 7). It shows that the rheological regulator (HSCN) prepared by the composite modifier has the best lipophilicity and optimal dispersion in the drilling fluid. The modifier of quaternary ammonium salt enters the montmorillonite interlayers and adsorbs on the surface, which significantly reduces the surface polarity of the montmorillonite and enhances the lipophilicity. In addition, the composite modification of amide substance and double-chain quaternary ammonium salt further increases the interlayer spacing of montmorillonite, which allows more oil molecules to enter into the interlayer of the crystals, and further improves the lipophilic property of the rheological regulator.

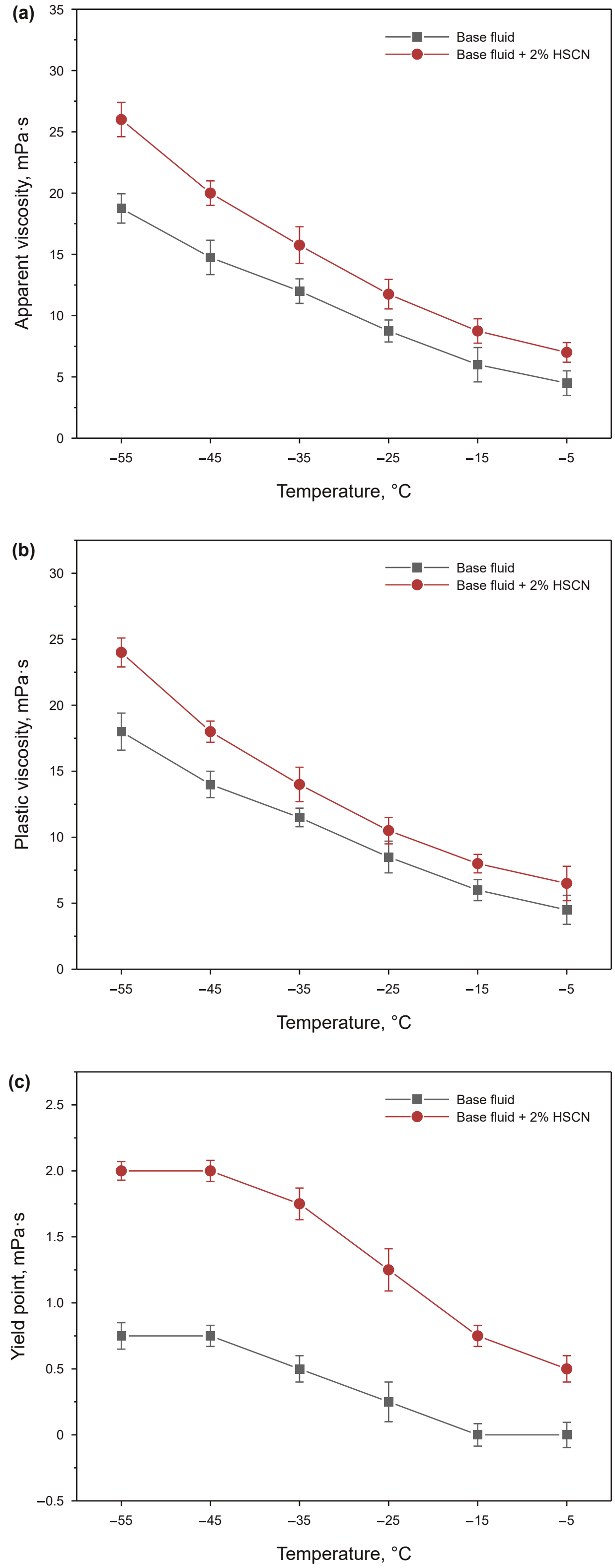

Fig. 14. Viscosity-temperature characteristics of drilling fluids ((**a**) Variation of apparent viscosity with temperature; (**b**) Variation of plastic viscosity with temperature; (**c**) Variation of yield point with temperature).

3.2. Evaluation of HSCN performance

3.2.1. Colloidal rate

3.2.1.1. Effect of different modifiers on colloidal rate of rheological regulators. The colloidal rate of the rheological regulators prepared by different modifiers at −55 °C was investigated separately, and the research outcomes are revealed in Fig. 8. The concentration of rheological regulators is 2%.

It was found that the colloidal ratio of the rheological regulator modified by CPL after 24 h was 22.4%, and that of the rheological regulators modified by DODMAC and DODMAC + CPL after 24 h was 81.6% and 90.4%, respectively. It shows that the rheological regulator (HSCN) modified by DODMAC + CPL has the best dispersion performance in polar drilling fluid. The reason for the analysis is that when the montmorillonite was modified with CPL alone there is no intercalation effect. It can not improve the structure of the montmorillonite crystalline layer, and the lipophilicity is poor, which results in a low colloidal rate in the oil. When the montmorillonite is modified with the composite modifier (DODMAC + CPL), the physical entanglement of the double-chain quaternary ammonium salt into the montmorillonite crystalline layer as well as the formation of hydrogen bonding between caprolactam and montmorillonite increase the spacing of the montmorillonite crystalline layer, and consequently more oil molecules enter into the interlayer of the montmorillonite lamellae, which results in the improvement of montmorillonite dispersibility and thus the maximum colloid rate. This is consistent with the findings of Sections 3.1.2 and 3.1.4.

3.2.1.2. Effect of rheological regulator (HSCN) concentration on colloid rate. It was concluded that rheological regulators were not easy to desorb after adsorption in the oil phase at low temperatures. However, low-temperature aggregation occurred, resulting in the deterioration of the rheological properties for drilling fluids. Accordingly, it is crucial to investigate the HSCN dispersion at ultra-low temperatures. HSCN was dispersed in the drilling fluid, and the colloid rate was evaluated at −55 °C. The findings are shown in Fig. 9.

It was studied that as the HSCN concentration increased, the colloid rate showed a constant improvement (Fig. 9). When the concentration of HSCN is 2%, the colloid rate is 90.4% after 24 h. It shows that HSCN has excellent colloidal properties under ultra-low temperature conditions.

3.2.2. Rheological properties

3.2.2.1. Effect of HSCN on viscosity. The influences of HSCN dosages on the viscosity were studied at −55 °C. The findings are revealed in Fig. 10.

From Fig. 10, as the HSCN concentration improves, the apparent viscosity and plastic viscosity gradually increase. This is because as the particle concentration of HSCN gradually increases, the network structure formed between particles becomes stronger, which in turn makes the viscosity increase.

3.2.2.2. Effect of HSCN on yield point and gel strength. The influences of HSCN dosages on the yield point and gel strength were studied at −55 °C. The findings are revealed in Fig. 11.

From Fig. 11, with the improvement of HSCN concentration, the yield point and gel strength have an increasing trend, and 2% HSCN can increase the yield point from 0.75 to 2.0 Pa. At the same time,

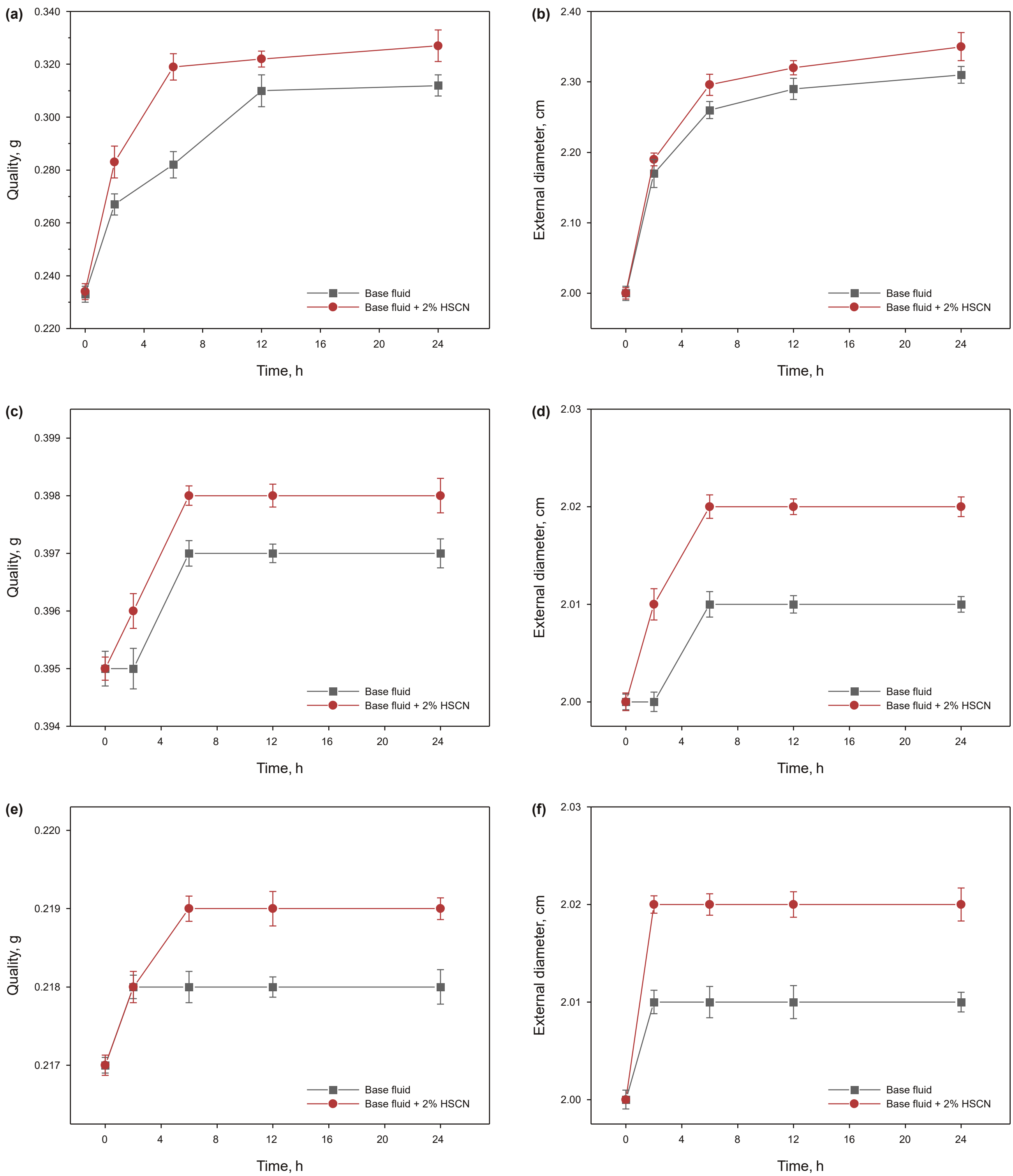

Fig. 15. Effect on rubber corrosion ((**a**) change in mass of silicone rubber; (**b**) change in outer diameter of silicone rubber; (**c**) change in mass of fluoroelastomer; (**d**) change in outer diameter of fluoroelastomer; (**e**) change in mass of Buna-N; (**f**) change in outer diameter of Buna-N).

the gel strength (10 min) of the drilling fluid is increased from 0 to 1.25 Pa. It shows that HSCN can effectively increase the yield point and gel strength, which can enhance the suspension and carrying abilities of ice debris and rocks cuttings.

In addition to the above studies, this section also compares the rheological properties of 1.5% HSCN and 1.5% Baker organoclay (commercial clay) at −55 °C, including apparent viscosity, plastic viscosity, yield point and gel strength. The experimental results are shown in Fig. 12.

It was shown that the viscosity of drilling fluid containing Baker organoclay was close to that of HSCN, but HSCN made the drilling fluid have higher yield point and gel strength than Baker

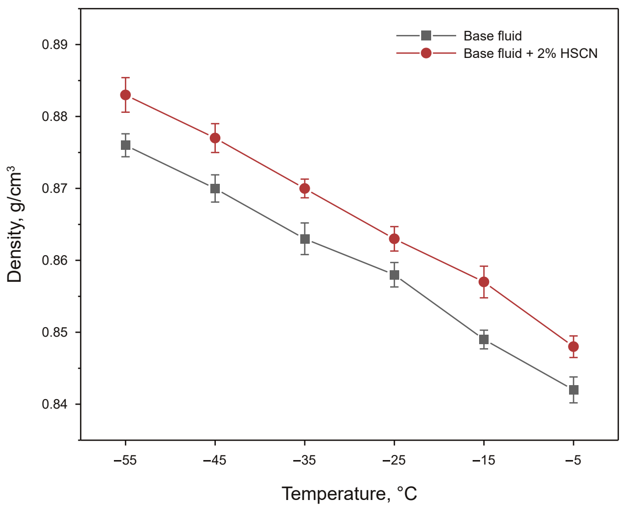

Fig. 16. Variation relationship of drilling fluid density with temperature.

Table 1
Zeta potential of base fluid and drilling fluid with 2% HSCN.

Samples	Zeta potential, mV	Standard deviation, mV
Base fluid	−2.36	0.62
Base fluid +2% HSCN	−10.1	0.98

organoclay. Therefore, the rheological properties of HSCN in drilling fluid are excellent.

3.2.2.3. Effect of HSCN on shear dilution performance. The effect of HSCN on the viscosity at different shear rates was investigated, and the shear dilution characteristics of drilling fluid were disclosed (Fig. 13).

For the base fluid, as the shear rate improves, the viscosity changes slowly. However, after adding HSCN to the base fluid, the viscosity reduces rapidly and changes obviously when the shear rate increasingly improves, providing outstanding non-Newtonian characteristics of shear dilution. When the drilling fluid shows the characteristics, it will own higher yield point and form the network structure, which can efficiently increase the suspension and carrying abilities of ice debris and rocks cuttings, and guarantee the wellbore cleanliness (Ajieh et al., 2023; Huang et al., 2023).

3.2.3. *Viscosity-temperature characteristic*

It is very important for rheology regulation of drilling fluids to investigate the variations of viscosity and yield point with temperature. In this section, the changes of rheology parameters for base fluid, drilling fluid with 2% HSCN were studied at temperatures from −5 to −55 °C, respectively (Fig. 14).

Compared with the base fluid, HSCN significantly improves the yield point, and the improvement of viscosity is controllable. Moreover, with the decrease of temperature, HSCN makes the yield point constantly improve, and the improvement of yield point is significant at different temperatures. It shows that HSCN can obviously enhance the viscosity-temperature characteristics for drilling fluid.

3.2.4. *Evaluation of rubber corrosivity*

Rubber was widely used in the Antarctic drilling process, such as sealing the pressure chamber of electro-mechanical drilling tools (Liu et al., 2016). However, the drilling fluid will inevitably corrode the rubber material when the drilling tool is in the drilling fluid for a long time, which may influence the properties of the drilling tools. Treatment agents are the core part of drilling fluid and determine the main properties of drilling fluid. Thus, it is of very importance to explore HSCN corrosion to rubber materials.

The corrosiveness of HSCN on different rubber was evaluated by testing the effects of base fluid and drilling fluid with 2% HSCN on the outer diameter and mass of different rubber over time under the condition of −55 °C, respectively. The experimental results are shown in Fig. 15.

It was found that, after the three different rubber were immersed in the base fluid and the drilling fluid with 2% HSCN for 24 h, the mass increase of silicone rubber was 0.079 and 0.093 g, and the increase of outer diameter was 0.31 and 0.35 cm, respectively; The mass increase of fluoroelastomer was 0.002 and 0.003 g, and the increase of outer diameter was 0.01 and 0.02 cm; The mass increase of nitrile rubber was 0.001 and 0.002 g, and the increase in external diameter was 0.01 and 0.02 cm, respectively. It was shown that HSCN was weakly corrosive to all three different rubber. Compared with the base fluid, the mass and outer diameter of both fluoroelastomer and nitrile rubber were only increased by 0.001 g and 0.01 cm, respectively, after immersion in drilling fluid with HSCN.

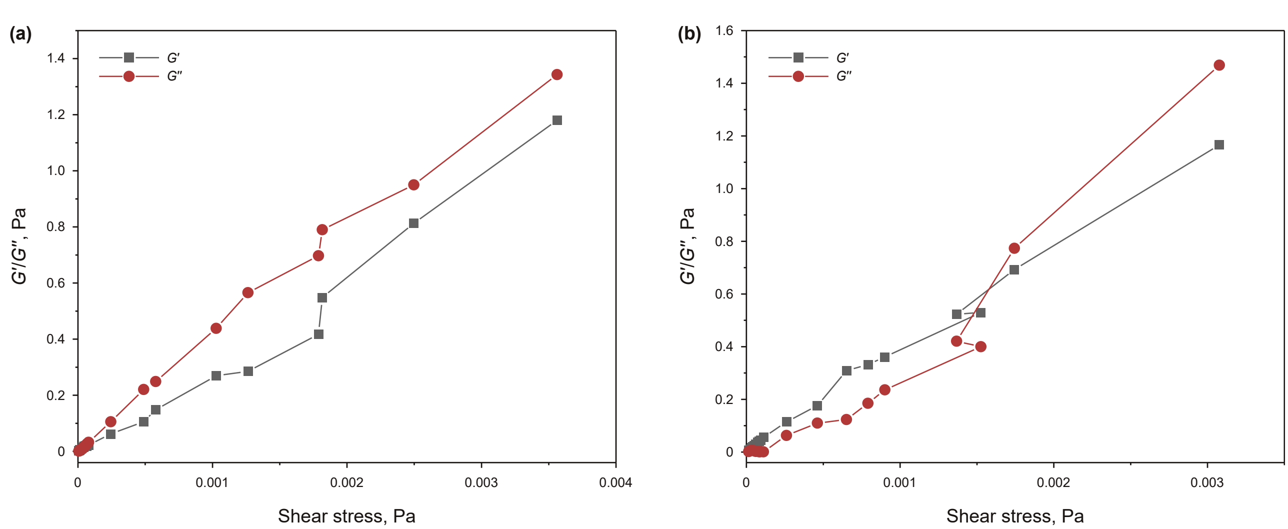

Fig. 17. Variation of drilling fluid modulus with shear stress ((**a**) base fluid; (**b**) base fluid +2% HSCN).

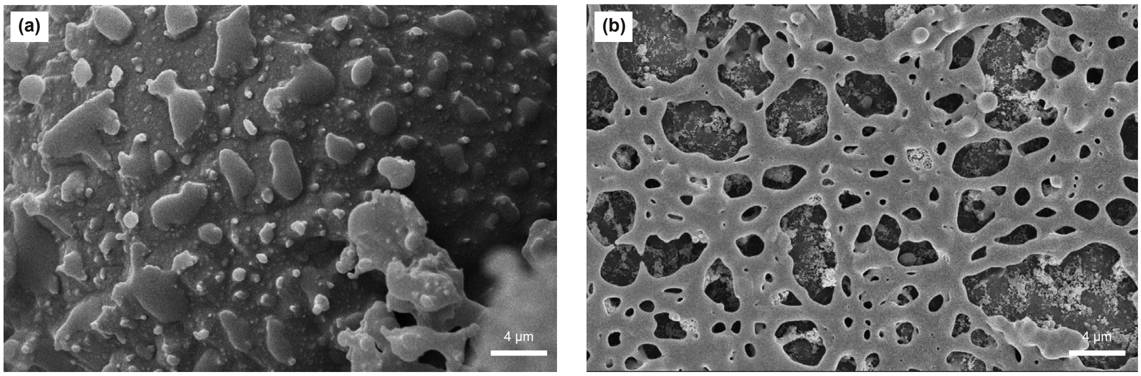

Fig. 18. Images of cryo-scanning electron microscopy ((**a**) base fluid; (**b**) base fluid +2% HSCN).

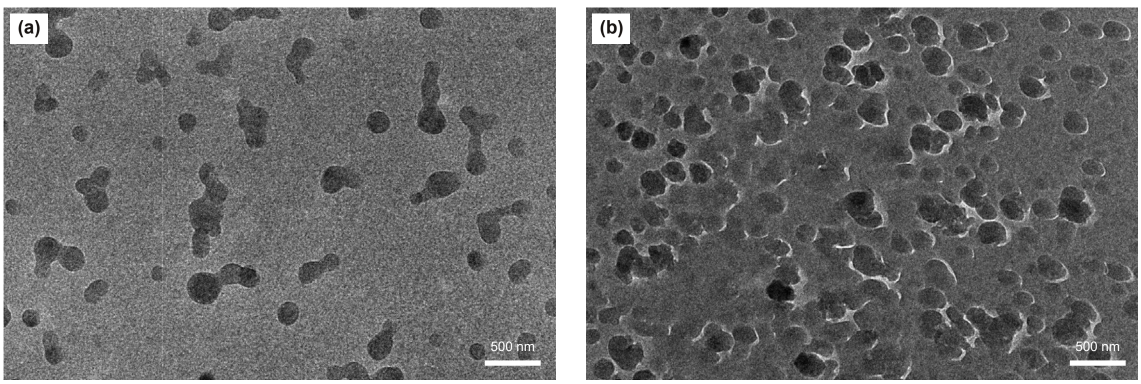

Fig. 19. Images of cryo-transmission electron microscopy ((**a**) base fluid; (**b**) base fluid+2% HSCN).

3.2.5. Density and zeta potential tests

In this section, the densities and zeta potentials of the base fluid and the drilling fluid with 2% HSCN were measured. The experimental results are shown in Fig. 16 and Table 1, respectively.

The study showed that the density of drilling fluid increased gradually with the decrease of temperature. And at −55 °C, the density of drilling fluid with 2% HSCN reached 0.883 g/cm^3.

From Table 1, compared with the base liquid, HSCN improves the Zeta potential of drilling fluid, indicating that the dispersion performance of HSCN in drilling fluid is excellent.

3.3. Mechanism research

3.3.1. Analysis of modulus

By testing the changes in modulus of drilling fluid with shear stress, the grid structure strength of drilling fluid was analyzed (Kim et al., 2003). The changes of elastic modulus (G') and viscous modulus (G'') with shear stress were investigated for base fluid and drilling fluid with 2% HSCN, respectively.

For the base fluid (Fig. 17(a)), as the shear stress increases, the viscous modulus (G'') is throughout greater than the elastic modulus (G'), showing that there is no grid structure in base fluid. For the drilling fluid with 2% HSCN (Fig. 17(b)), the elastic modulus is greater than the viscous modulus when the shear stress is lower than about 1.58×10^{-3} Pa, while the viscous modulus starts to be greater than the elastic modulus when the shear stress is more than 1.58×10^{-3} Pa. The findings indicate that the grid structure of drilling fluid is damaged by shear stress of about 1.58×10^{-3} Pa. This also shows that HSCN can effectively enhance the structure strength of drilling fluid, and thereby improve the rheology performances.

3.3.2. Cryo-scanning electron microscopy

The microstructures of base fluid and drilling fluid with 2% HSCN were investigated by cryo-SEM. The microstructures are revealed in Fig. 18.

The drilling fluid with HSCN shows a continuous "honeycomb" grid structure (Kim et al., 2003) (Fig. 18(b)). This is the result of the physical adsorption and winding between the long carbon chains of HSCN, as well as the interaction between polar groups, which together form a grid structure. The appearance of this structure improves grid structure strength of drilling fluid, leading to an improvement in the yield point, as well as an enhancement in the shear dilution performance of the drilling fluid, which ultimately improves the rheology performances. However, a similar structure did not appear in the base fluid (Fig. 18(a)).

3.3.3. Cryo-transmission electron microscopy

In order to study the rheological regulation mechanism of HSCN more deeply, the internal structure of the drilling fluid was analyzed by cryo-TEM.

From Fig. 19, HSCN generates inter-particle interactions in the drilling fluid, and these interactions are dense (Fig. 19(b)). The analysis is due to the electrostatic attraction and hydrogen bonding interactions between HSCN particles forming a mesh structure, resulting in dense interactions in the drilling fluid. The formation of this structure effectively enhances the shear force, increases the suspension and carrying abilities of ice debris and rocks cuttings for drilling fluid, and also enhances the colloidal stability of drilling fluid.

3.3.4. Mechanism analysis

In order to better elaborate the role of HSCN in improving the

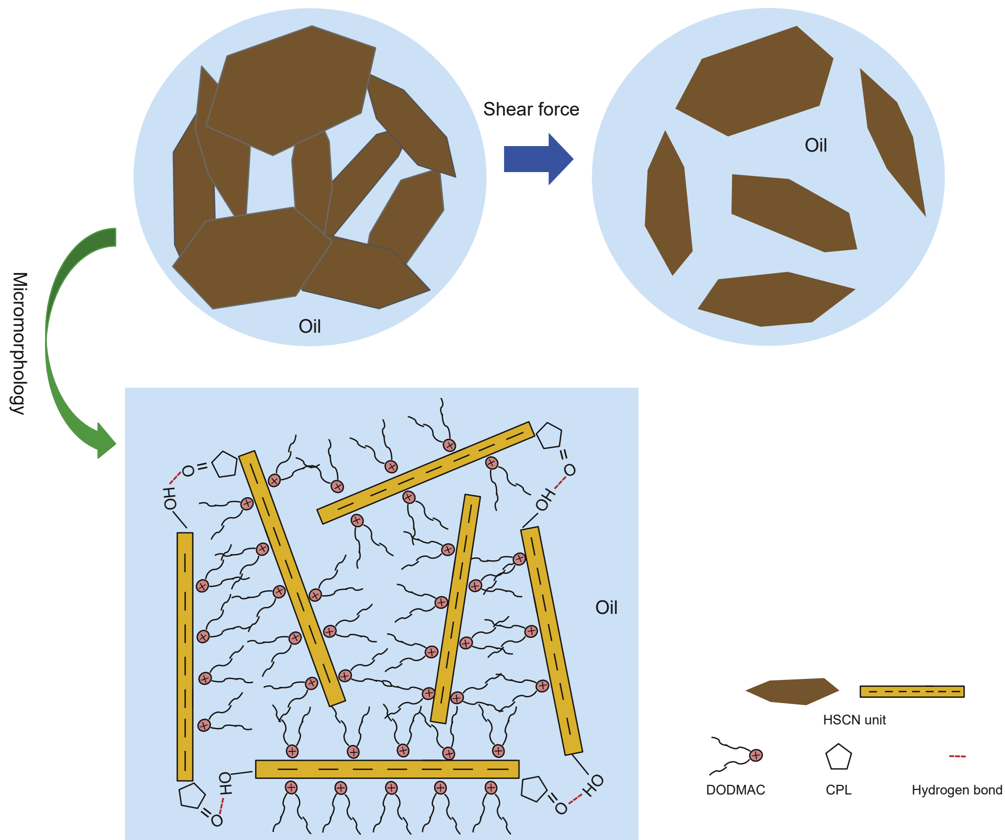

Fig. 20. Schematic diagram of the mechanism of HSCN to improve rheological properties.

rheology performances of drilling fluids, the proposed mechanism is shown in Fig. 20.

The action mechanism of rheological regulator (HSCN) is that the staggered laps between HSCN lamellar units form an infinitely extended reticular structure. The structure is mainly formed by electrostatic attraction between HSCN particles, hydrogen bonding, physical adsorption and entanglement between long carbon chains in HSCN molecules. At the same time, the high crystal layer spacing of HSCN allows more oil molecules to enter the interlayer of HSCN crystals, which further improves the dispersion of HSCN molecules in the drilling fluid and efficiently enhances the rheology performances. Moreover, when an external force (shear force) is applied, the network structure of HSCN molecules in the drilling fluid is disrupted, and the lamellae of connected HSCN units are separated, leading to a decrease in the viscosity of the drilling fluid, which in turn, HSCN exhibits excellent shear dilution properties.

However, the main objective of this study was the drilling of the Antarctic ice sheet, and the drilling of the rock formations under the ice sheet was not considered enough. In the future, it is necessary to study the performance of drilling fluid in rock formations, including the plugging performance and filtration performance of drilling fluid under low temperature conditions.

4. Conclusions

In this study, a rheological regulator (HSCN) of ultra-low temperature drilling fluid was synthesized by composite modification of MMT with DODMAC and CPL. The X-ray diffraction test indicated that HSCN layer spacing was 1.833 nm, and the modifier successfully entered the montmorillonite interlayer. Scanning electron microscopy analysis shows that compared to montmorillonite, HSCN contains more stripped clay layers and significantly reduced particles. The contact angle analysis shows that the contact angle of montmorillonite is 46.2°, and the contact angle of HSCN is 14.1°. This indicates that HSCN has excellent lipophilicity. The HSCN performance test showed that 2% HSCN could maintain the colloid ratio of the drilling fluid to 90.4% after 24 h at −55 °C, with the yield point of 2.0 Pa, and has excellent shear dilution non-Newtonian characteristics. Mechanism study shows that the staggered overlap between HSCN layer units forms an infinitely extended reticular structure. The structure is mainly formed by electrostatic attraction between HSCN particles, hydrogen bonding, physical adsorption and entanglement between long carbon chains in HSCN molecules. The rheological regulator (HSCN) has excellent colloidal properties, rheological properties and low corrosive properties at ultra-low temperatures, providing new insights into enhancing the rheology performances of ultra-low temperature drilling fluids, and has potential applications in Antarctic drilling projects. However, this study mainly focused on drilling in ice sheets, while drilling of the bedrock under the ice was not sufficiently considered. Therefore, further research will be carried out in the future research process.

CRediT authorship contribution statement

Ning Huang: Writing – original draft, Visualization, Methodology, Investigation, Conceptualization. **Jin-Sheng Sun:** Validation, Supervision, Project administration, Conceptualization. **Jing-Ping Liu:** Validation, Resources, Project administration, Funding acquisition. **Kai-He Lv:** Writing – review & editing, Supervision, Investigation. **Zong-Lun Wang:** Visualization. **Xue-Fei Deng:** Investigation, Data curation. **Zhi-Wen Dai:** Visualization. **Xian-Fa Zhang:** Investigation, Data curation.

Declaration of competing interest

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.

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Abbreviations

DODMAC	Dimethyl dioctadecyl ammonium chloride
CPL	Caprolactam
HSCN	Name of rheological regulator
TEM	Transmission electron microscopy
SEM	Scanning electron microscopy
XRD	X-ray diffraction
MMT	Montmorillonite
AR	Analytical purity

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