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Research of Na_2CO_3 / $C_{12}H_{25}SO_4Na$ modified bentonite on high dispersion performance

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ABSTRACT

The dispersion performance of drilling fluids at high temperatures has always been a bottleneck in the research. To improve the dispersion of drilling fluid at high temperature and pressure, the natural Ca-based bentonite was modified with Na_2CO_3 and SDBS (sodium dodecyl benzene sulfonate: $C_{12}H_{25}SO_4Na$) by microwave semi-dry method. The effects of the ratio of Na_2CO_3 to SDBS, radiation time, initial temperature and microwave power on the modification were investigated. In addition, the microstructure of the samples was obtained using infrared spectroscopy (FTIR) and scanning electron microscopy (SEM), and the results showed that Na_2CO_3/SDBS was successfully inserted into the bentonite. In addition, the dispersion performance of the modified bentonite was evaluated in this paper, and the gelling value of the modified bentonite was 525 ml/15 g with an apparent viscosity of 22.5 mPa·s. The modified bentonite was compared with drilling-grade Na-based bentonite. The fluid loss (FLHTHP) of modified bentonite at high temperature and pressure is only 8 mL, the mud productivity is up to 25.71 m^3/t, and the kinetic plastic ratio of modified bentonite is 1.5. The modified bentonite has better dispersion performance at high temperature and pressure.

Keywords: Bentonite; Anionic surfactants; Dispersion; Drilling fluid; Modification.

1. INTRODUCTION

The use of surfactants to modify bentonite is a major issue in the development of oil and gas resources. Engineers have done much research to prevent formation damage, reduce fluid loss of drilling fluid and keep formation stability [1]. However, the dispersion performance of drilling fluids at high temperatures has always been a bottleneck in the research [2]. Bentonite, as an important additive in drilling fluid, has a great influence on its rheological properties and filtration performance. However, natural bentonite cannot be directly used for drilling, so modification of bentonite has become a hot topic in recent years.

KUMARARAIA, *et al.* [3] Modified Bentonite with the cationic surfactant hexadecyl trimethyl ammonium bromide (HTAB) as landfill liner to retard the transportation of bisphenol A (BPA) for the first time, which showed that the HTAB-bentonite took on its notable advantages as components of landfill liners material to retain BPA in leachate. And Na-based bentonite was modified with HTAB. Because many quaternary ammonium cations were adsorbed on the surface of HTAB-bentonite, a decrease of the contact angle was found, which suggested an increasing affinity for oil, besides the rheological properties of the bentonite fluid easily affected by temperature [4]. When YI, *et al.* [5] modified clay with nonionic surfactants, they found that the advantage of nonionic surfactants over ionic ones is that they improve the thermal and chemical properties of organoclays, and are also more readily biodegradable. The study indicated that the organic bentonite modified by nonionic surfactants was the better choice for drilling fluids. Nano-particles of bentonite was prepared by nano-grinding, its performance was evaluated, by RATLCIEVICIU, *et al.* [6] The index of evolution included rheological properties, fluid loss, and gel strength, and it was treated by viscosifying agent and fluid loss agent, and compared with API-compliant bentonite, with decreasing the grain size of bentonite to the nano-scale, which could not meet the API-standard. By comparing the effects of cellulose nanocrystals (CNCs) and cellulose nanofibres (CNF) on the rheology and filtration of modified bentonite, Song *et al.* showed that both CNCs and CNF can reduce the viscosity of bentonite, and RIAZ, *et al.* [7] found that CNCs bentonite can form a filter cake with lower perme-

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ability. At present, bentonite used in drilling fluid is mainly Na-based bentonite (Na-Bent) or organic bentonite, which is unable to meet the overall drilling engineering requirements. The rheology (viscosity) of Na-bentonite suspension is easily affected by temperature. When the temperature rises, the viscosity of drilling fluid changes greatly, which easily causes well collapse. XU, *et al*. [8]. prepared intelligent temperature-sensitive bentonite (NIPAM-B) by grafting N-isopropyl acrylamide (NIPAM) onto the surface of sodium-based bentonite through the dehydration condensation of silane coupling agent KH570, in which the rheology of the modified bentonite was relatively stable, with a slight decrease in suspension within a controlled range. Liao *et al* [9]. synthesized a hydroxyethyl cellulose-acrylamide graft polymer by microwave-assisted synthesis and prepared bentonite composites based on this graft polymer. A minimum fluid loss of only 9.3 ml was prepared, which can meet the requirements of drilling grade bentonite. Liang *et al* [10], researched the dispersion properties of acidified bentonite in the aqueous phase. The article pointed out that improving the dispersion performance of bentonite can significantly improve the specific surface area and pore structure, increase the electrostatic repulsion and spatial site resistance due to adsorption of dispersant, reduce the aggregation and flocculation between particles, so the dispersion in the best state of dispersion, the smallest settling volume and the lowest flow viscosity.

Although modified bentonites such as Na-Bent and organic bentonite have been used in drilling fluids in related studies, the dispersibility at high temperatures still needs to be improved. There is currently only research in our laboratory that anionic surfactants and inorganic sodium salts combined to modify natural calcium-based bentonite (Ca-Bent) for use in drilling fluids. The sodium modification of calcium-based bentonite has improved its caking and suspension properties and we have published the paper. SUN, *et al*. [11] obtained modified substances with high viscosity properties by compound modification of bentonite. It was applied to drilling fluids to improve the performance of drilling fluid applications. The surfactant sodium dodecyl benzene sulfonate (SDBS) ion slows down the coalescence of clay particles at high temperatures, it forms a water film and adsorbs to the surface of the clay particles, providing a stabilizing effect.

Because of the poor dispersion ability of bentonite at high temperatures and pressures. The study aimed to further investigate the dispersion performance of modified bentonite at high temperature and high pressure by testing the gelling value (this property is an important indicator of dispersibility) by modifying natural Ca-Bent with Na_2CO_3 and SDBS by microwave semi-dry method. In addition, the fluid loss at high temperature and pressure (FL_{HTHP}) was tested in this study to compare the Na_2CO_3 / SDBS modified bentonite with drilling-grade Na-based bentonite from an oil field for practical use.

2. MATERIALS AND METHODS

2.1. Materials and equipment

The following materials and instruments were used for the experiments: The natural Ca-Bent (325 mesh) for preparing the modified bentonite was supplied by Lingshou County Xing Yuan mineral powder processing plant (Hebei, China); The basic information of bentonite is as follow: swelling volume 18 ml, pH = 6, montmorillonite content near 85 %, colloid index 150 ml/15 g and density 6 g/cm^3, etc.; sodium carbonate, SDBS and absolute ethanol (analytical reagent) from Sinopharm Chemical Reagent Co, Ltd. (Shanghai, China) were used in this study. The drilling-grade bentonite comes from commercially available bentonite.

2.2. Method

2.2.1. Purification

The natural Ca-Bent samples were sublimated before using in the experiments as follows. Ca-Bent (325-mesh) were mixed with distilled water, then the mixtures were stirred for 30 min. After stirring, all mixtures were stood for 1 h and the small impurities suspended in the supernatant were removed with a pipette. This process was repeated until the supernatant was clear. Finally, the resulting products were then dried at 105 °C. After drying, the product was ground and sieved to obtain a 300-mesh powder.

2.2.2. Modification

Firstly, five samples of 1 g Na_2CO_3 were weighed, then 0 g, 0.5 g, 1 g, 1.5 g, 2 g SDBS were weighed separately. Afterward, Na_2CO_3 was mixed with SDBS. Then each chemical was dissolved in 50% anhydrous ethanol to obtain the desired modifier. After that 5 g of natural Ca-based bentonite was taken and mixed with the modifier and the mixture was made into the semi-dry reaction material. The mixture was placed evenly on the bottom of the flask and kept still for 30 minutes before microwave treatment. Then, the flask with the reaction material was put into microwave apparatus and different parameter of the microwave was set. The parameters of the microwave included time, initial temperature, and microwave power. Especially the time ranged from 1 min to 5 min, and the temperature ranged from 30 °C to 70 °C (room temperature 30 °C), and the power was from 400 W to 800 W. Moreover, the electromagnetic stirring speed should always be controlled at 500 r/min. After, the reaction material was placed in a digital display blast drying box and dried at 80 °C. Finally, the reaction material was ground and sieved so that 140-mesh modified bentonite powder is obtained for the test.

2.2.3. Characterization

Fourier transforms infrared (FTIR) analyses were performed at room temperature in the spectral range of 4000 – 400 cm^{-1} using an FTIR spectrometer. The experimentally obtained modified bentonite and drilling-grade Na-based bentonite were mixed and ground thoroughly with dewatered KBr, respectively, and the mixture was made into a pellet by a hydraulic press. The spectra were collected with 64 scans of accumulation at a resolution of 4 cm^{-1}. Scanning electron microscope (SEM) photos were taken at room temperature in the focus of 10 m, accelerating voltage of 15 kV, and magnification of 5000 using a field emission electron scanning microscope.

2.2.4. Measurement

According to the Chinese Standards of Bentonite [12], the gelling value was tested. Besides, the apparent viscosity and yield point and plastic viscosity ratio of modified bentonite and drilling-grade Na-based bentonite were measured according to the Specification for Drilling Fluid Materials [13].

To obtain the slurry rate of bentonite slurry, the following operations are carried out. Firstly, the bentonite quality required for preparing 15 mPa·s bentonite solution was measured. And the specific gravity of bentonite suspension was measured by liquid densimeter. The formula (2) for calculating the rate of bentonite slurry is as follows:

$$B = \frac{V_w}{W_x} + \frac{1}{M_x} \tag{1}$$

Where B = pulping rate (m^3/t), V_w = water volume (ml), W_x = weight (g), M_x = specific, gravity(g/cm^3).

Then, 22 g of modified bentonite was mixed with 400 ml of distilled water to obtain the modified bentonite suspension. And the mixture was stirred under 1000 r/min, which spent 20 min, and then mature 24 h, afterward stirred 5 min again under 1000 r/min. In the same way, the drilling-grade Na-based bentonite was made into suspension. Next, the two suspensions were poured into the reactor, and the reactor is placed in a heating furnace, after retrogradation of the bentonite at 90 °C, 120 °C, 150 °C, 180 °C, 210 °C for 16 h, respectively. Then, FL_{HTHP} was measured, and the stability of the bentonite was analyzed after high temperature treatment.

Finally, 2 g of modified bentonite was mixed with 35 ml of distilled water were taken, meanwhile, the mixtures were shaken well in the cylinder with a bottle plug so that the bentonite completely dispersed in distilled water, and kept 24 h, then the suspended situation was observed. The suspension of drilling-grade Na-based bentonite was observed in the same way.

3. RESULTS

3.1. Optimization of preparation conditions for Na_2CO_3/SDBS bentonite

In the preparation of modified agent, the work will fix the amount of sodium carbonate, and gradually increase the amount of SDBS, then the ratio of Na_2CO_3 to SDBS changed. It can be seen from Figure 1(a) that the gelling value of bentonite increased sharply with the increase of SDBS. It is presumed that the special layered structure is enlarged by the modified agent. When the ratio of Na_2CO_3 to SDBS was 1: 0.5, the gelling value of bentonite reached the high value of 450 ml/15 g. And with the amount of SDBS increased, the gelling value of bentonite quickly decreased, then its tendency was gentle. Finally, the gelling value of bentonite suddenly decreased. There is no more space for inhaled water molecules because sodium ions and organic ions have occupied the interlayer of bentonite, which results in the decrease of the hydration degree. Finally, the optimum ratio of Na_2CO_3 to SDBS was chosen as 1:0.5.

It can be seen from Figure 1(b) that the starting point 30°C is room temperature, and the gelling value of bentonite increased reposefully with the increase of initial temperature. When the initial temperature of the microwave is 60 °C, the greatest gelling value of bentonite is 450 ml/15 g. Then the gelling value declines rapidly. The trend is contributed to temperature, with the initial temperature is progressively raised, and the reaction is more intense. It is SDBS and Na_2CO_3 lead to well colloidal property of bentonite, because Na^+ embed in

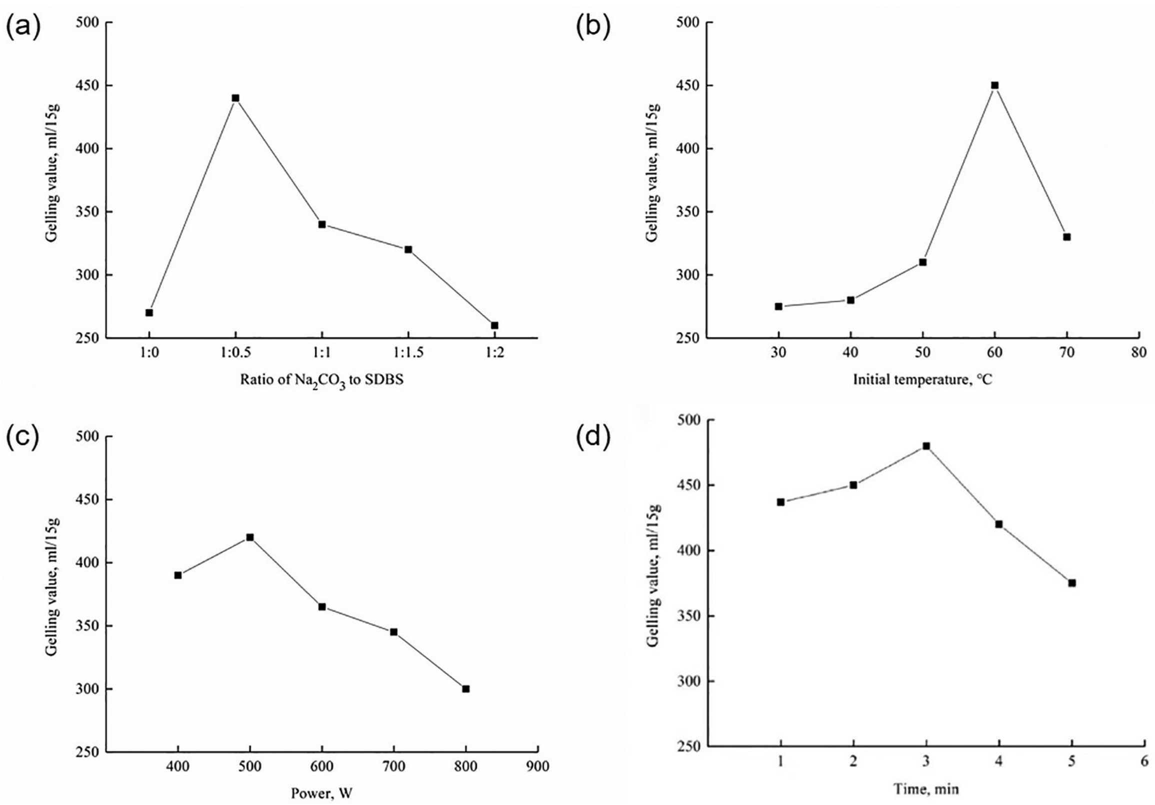

Figure 1: Optimization of preparation conditions for Na_2CO_3/SDBS Bentonite: (a) Ratio of Na_2CO_3 to SDBS, (b) Initial temperature, (c) Power, (d) Time.

SDBS is contributed to micellar growth [14]. However, the balance is broken under temperature. So the superb temperature is 60 °C.

It can be seen from Figure 1(c) that the power increased from 400 W to 500 W, the gelling value increases sharply from 390 ml/15 g to the maximum value of 420 ml/15 g. Moreover, after a short period of stability, the gelling value quickly reduced again. It may be the expanding effect of microwave leads to the increase of the spacing of bentonite, the effect that refers to the penetration of microwave energy into the material and the expansion of the internal organization of the material being processed. This leads to the reinsertion of reactant molecules in a certain power range [15]. The gelatinous valence decreases sharply at a power greater than 500 W. This is because the irradiation intensity becomes stronger, which makes the SDBS carbon chains break and disrupts the normal proceeding of the reaction.

As shown in Figure 1(d), the gelling value rose slowly at the beginning as the microwave time increased. When the microwave time was 3 min, the maximum gelling value of bentonite was 480 ml/15 g. Therefore, it can be assumed that the energy of the reaction was insufficient at the beginning, and when the time was longer, the reactants were fully reacted. Thereafter the curve suddenly decreased with a slow decreasing trend. This may be due to the fact that the temperature of the reaction increases sharply with the extension of time, leading to strong molecular thermal motion, which may be the result of the change of the surface at the beginning. If the time continues to be extended, the ion exchange equilibrium reaction will be broken. Therefore, a reasonable time is set at 3 min.

To sum up, the reasonable condition was the ratio of Na_2CO_3 to SDBS 1:0.5, the initial temperature of 60 °C, the power of 500 W, and the reaction time of 3 min.

3.2. Characterization of bentonite and its mechanism

3.2.1. FTIR spectra

Figure 2 shows the FTIR spectra of the modified bentonite by SDBS-Na_2CO_3 (spectrum a) and unmodified bentonite (spectrum b). The FTIR spectra of Figure 2(b) display broadband around 3627 cm^{-1} which corresponds

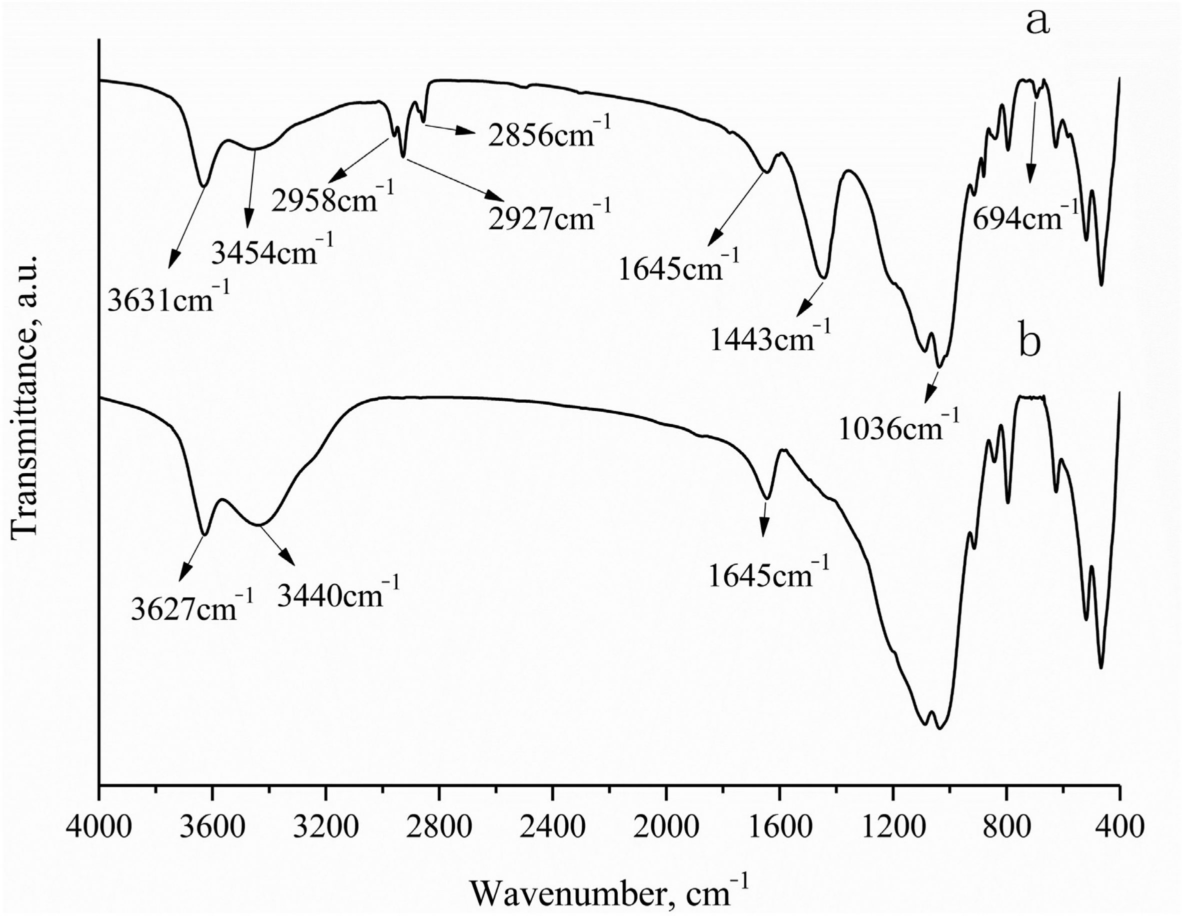

Figure 2: FTIR spectra of the modified bentonite by SDBS-Na_2CO_3 (spectrum a), drilling-grade Na-based bentonite (spectrum b).

to the Al—OH stretching vibration of the hydroxyl, and the peak of Si-O-Si is around 1036 cm^{-1}. In the FTIR spectra of Figure 2(a), the above two main absorption peaks did not change, indicating that the bentonite silicate skeleton did not change. Besides, new peaks appearing around 2856 cm^{-1}, 2927 cm^{-1}, 2958 cm^{-1} are due to —CH_2—, —CH_3 stretching and bending vibration on SDBS. The characteristic absorption of —SO_3— vibration is also evidenced around 694cm^{-1}. The peak around 1443 cm^{-1} is associated with the skeleton vibrations of the benzene ring skeleton, which is due to the C—H stretching vibration in the benzene ring backbone. The absorption peak at 1645 cm^{-1}, representing H—O—H, is significantly lower in Figure 2 (a) than in the spectrum b, which indicates that the adsorbed water between the bentonite layers is extruded by SDBS, and the SDBS is successfully intercalated in the bentonite [16]. In addition, a small amount of SDBS is adsorbed on the surface of bentonite due to van der Waals force, and the hydrophilic group of —SO_3— is away from the bentonite, and the alkyl moiety is close to the bentonite.

3.2.2. SEM

It can be seen from Figure 3(a) is the SEM picture of a natural Ca-Bent. It is shown that the montmorillonite layer in the original natural Ca-Bent is compact, flat and contains some non-montmorillonite impurities. Figure 3(b) is the SEM picture of the drilling-grade Na-based bentonite, its shape presented as flower-shaped floc, these flocs mutually connect into a distinct network structure, and sometimes these flocs like petal-shaped slices. Figure 3(c) is Na_2CO_3 modified bentonite, it was similar to SEM of the drilling-grade Na-based bentonite. From Figure 3 (d), It can be seen that the montmorillonite layers are looser than other soil layers. Most of the lamellae are curled and pores appear between the curled layers, which may be due to bentonite adsorb different cations. The morphological feature of composite modified soil will increase the utilization ratio of additives in drilling fluid preparation, thus improving drilling fluid performance [17].

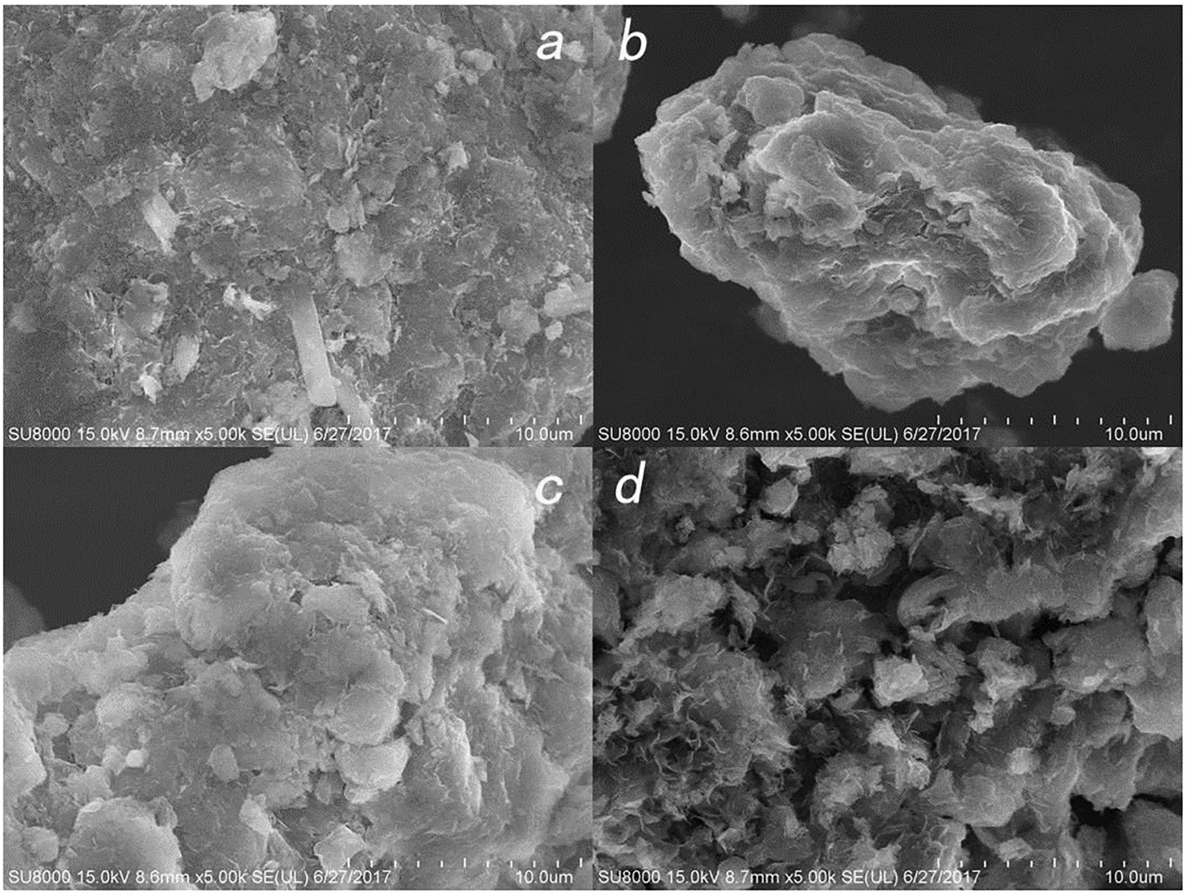

Figure 3: SEM of bentonite: (a) Original Ca-Bent, (b) drilling-grade Na-based bentonite, (c) Modified bentonite by Na_2CO_3, (d) Modified bentonite by SDBS and Na_2CO_3.

4. DISCUSSION

The SDBS/Na_2CO_3-bentonite (SDBS/Na_2CO_3-Bent) was modified by SDBS and Na_2CO_3 under optimum conditions. Through the basic performance test of the two bentonites, it can be seen that the apparent viscosity of the two bentonite suspensions is the same 22.5 mPa·s, but the modified bentonite has a yield point and plastic viscosity ratio of 1.5 and the drilling-grade Na-based bentonite is only 0.88. A higher yield point and plastic viscosity ratio makes it easier for drilling fluids to carry the cuttings.

And the pulping rate of modified bentonite is as high as 25.71 m^3/t, which is higher than 10.51 m^3/t of drilling-grade Na-based bentonite. In addition, the measurement of the fluid loss of the two bentonites at high temperatures is as follows.

From Table 1, the FL_{HTHP} rose with increasing temperature, and when the temperature was 90 °C and 120 °C, the FL_{HTHP} of both were less than 16 ml. The temperature was more than 120 °C, the FL_{HTHP} of Drilling-grade Na-Bentonite was more than 16 ml. However, the FL_{HTHP} of modified-Bent was less than or equal to 16 ml in the range of 90–180 °C. Besides, when the temperature was 180°C, modified-Bent meets the requirements of drilling fluid. Moreover, the modified-Bentonite was more stable than drilling-grade Na-Bent in the scope of 90 °C to 210 °C.

From observing the suspension of modified bentonite and drilling-grade Na-based bentonite after sufficient dispersion, from Figure 4, it can be seen that after 12 h the modified bentonite and drilling grade bentonite remain in suspension, and after 24 h the drilling-grade bentonite starts to settle and white precipitation appears at the bottom of the measuring cylinder. In the drilling site, usually in the circulation of drilling fluid before the preparation of drilling fluid, after the completion of the preparation, to stand for 24 h before pumping into the formation, so to ensure that the drilling fluid 24 h to maintain suspension is the basic requirement for the preparation of efficient drilling fluid. Therefore, modified bentonite has better suspension. This is because the reaction process generates a large number of liquid beads in the suspension due to the adhesion of SDBS on the bentonite surface, which produces the Jamin effect. This effect is due to the fact that the generated liquid

Table 1: The results of temperature resistance of modified bentonite and drilling-grade Na-based bentonite.

Project (T/°C)	**Drilling-grade Na-based bentonite (FLHTHP/mL)**	**Modified-Bentonite (FL_{HTHP}/mL)**
90°C	14.0	10.0
120°C	14.0	14.0
150°C	18.0	16.0
180°C	24.0	16.0
210°C	26.0	18.0

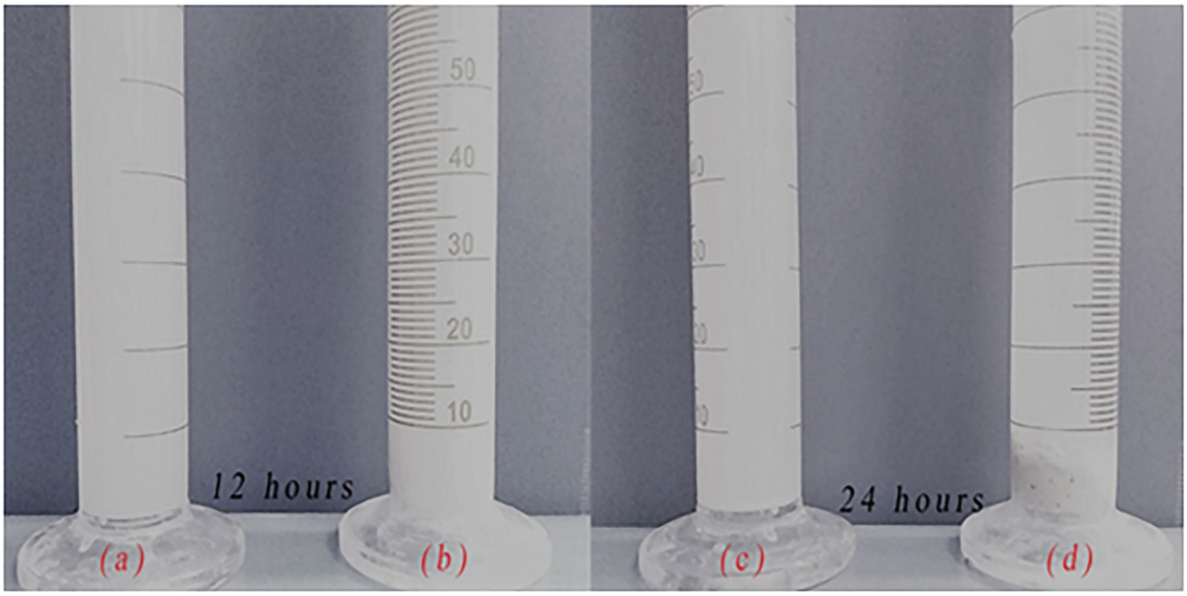

Figure 4: Suspension test of bentonite suspensions (a,c: modified bentonite; b,d: drilling grade Na-based bentonite).

beads reduce the leakage pore space to stop the filtrate from leaking to the lower layer, thus controlling the filtrate of the working fluid [18].

5. CONCLUSIONS

Anionic surfactant SDBS and inorganic salt Na_2CO_3 were first used to composite modified bentonite by microwave semi-dry method. By optimizing the synthesis conditions of SDBS/Na_2CO_3 bentonite, the best conditions were a ratio of SDBS to Na_2CO_3 0.5:1, microwave time 3min, microwave initial temperature 60 °C, and microwave power of 500 W.

From SEM, it is clear that the surface of the bentonite modified by SDBS and Na_2CO_3 was more uneven, showing a fine irregular curled sheet structure. From FTIR spectra, it is obvious that the intercalation agent successfully entered the layer of bentonite, and little adsorbed on the surface of the bentonite.

Under the best conditions, the performance parameters of the modified bentonite were better than the drilling-grade Na-based bentonite, as shown by the modified bentonite gelling value of 525 ml/15 g, apparent viscosity of 22.5 mPa·s, mud yield of up to 25.71 m^3/t, and yield point and plastic viscosity ratio of 1.5.

The modified bentonite can maintain low fluid loss at high temperature and high pressure, and the FL_{HTHP} was only 8 ml, so the bentonite has better temperature resistance. In addition, Na^+ of Na_2CO_3 opens up the interlayer space for $C_{18}H_{29}O_3S^-$, SDBS strengthen the surface properties of bentonite by the effect of Na_2CO_3. Besides, the dispersibility of modified bentonite is contributed to match with other drilling additives. The SDBS/Na_2CO_3 bentonite could reduce fluid loss and improve pulping performance and make drilling more efficient.

6. ACKNOWLEDGEMENTS

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