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# Dispersive and filter loss performance of calcium carbonate nanoparticles in water for drilling fluid applications

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### Abstract

Adding nanoparticles in a drilling fluid can aid in the sealing of the nanopores in the borehole wall rock and the mud cake; in this way, the filtrate loss of the drilling fluid can be reduced and the borehole wall is stabilized. In this work, the spectrophotometric method was used to study the effect of dispersants on calcium carbonate nanoparticles. The best dispersion effect was achieved at cetyltrimethyl ammonium bromide (CTAB) concentration of 4 wt.%, dispersing time of 45 min, pH value of 8 and stirring speed of 900 rpm. The structure analysis showed that the adsorption layer was formed on the surface of calcium carbonate nanoparticles after CTAB modification, and no new crystalline compounds appeared. Under these optimized dispersing conditions, aggregation was prevented as manifested by the dramatically decreased average particle size of calcium carbonate nanoparticles while the surface hydrophilicity and Zeta potential of calcium carbonate nanoparticles both increased. Furthermore, we showed that a drilling fluid incorporating such well dispersed calcium carbonate nanoparticles exhibit decreased filter loss and thus better performance in sealing compared to the calcium carbonate nanoparticles without dispersants.

Keywords: calcium carbonate nanoparticles; dispersion; drilling fluid; filter loss

### 1. Introduction

In the process of oil and gas well development, drilling fluids play an important role in transporting drilling chips, keeping drilling bits cool, reducing friction and balancing formation pressure. The properties of drilling fluids affect the efficiency of oil and gas well extraction<sup>[1]</sup>. Recent years, nanomaterials have been widely used in the development of oil and gas field. Various nanoparticles have been used to improve the performance of drilling fluids, such as silica<sup>[2,3]</sup>, graphene oxide<sup>[4-6]</sup>, nano-cellulosic materials<sup>[7]</sup>, and polymer nanoparticles<sup>[8]</sup>. Bai et al.<sup>[9]</sup> synthesized expandable composite microspheres SECMs by grafting AMPS and Amon the surface of the modified nano-SiO<sub>2</sub>, which can effectively plug the cracks in the mud cake. Mehran et al.<sup>[10]</sup> synthesized TiO<sub>2</sub>/polyacrylamide nanocomposite additive, and the laboratory research results show that the additive can improve the viscosity of mud cake and reduce the filtration loss. Bai et al. <sup>[11]</sup> used methacrylate (MMA) as monomer to synthesize polymethyl methacrylate emulsion nanoparticles by microemulsion polymerization. The experimental results show that polymethyl methacrylate emulsion has a strong inhibitory effect on shale expansion. The use of nanoparticles in drilling fluids can decrease filter losses<sup>[12,13]</sup> and enhance their rheological characteristics<sup>[14,15]</sup>. Nanoparticles with the right size can seal the tiny nanoscale holes of shale<sup>[16-20]</sup>. The

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### 2.1 Materials

Calcium carbonate nanopowder (50 nm) was purchased from Ruicheng Huana NanoMaterials Technology Co. (China). The main dispersants (AR) were prepared by ChengDu Kelong Chemical Co. (China), including CTAB, sodium dodecyl sulfate (SDS), sodium carboxymethylcellulose (CMC) and tween-80.

### 2.2 Preparation of calcium carbonate nanopowder suspension

Calcium carbonate nanopowder was added into the deionized water at a weight ratio of 8:100, and then the suspension was ultrasonically vibrated using an ultrasonic generator (Scientz-1200E, Ningbo Scientz Biotechnology Co., China) for 1 h at the frequency of 40 kHz. Then, the calcium carbonate nanopowder suspension was placed in the flask and under certain stirring speed and heated to 45 °C. Finally, to obtain different calcium carbonate nanoparticles dispersion systems, the dispersants, including CTAB, SDS, CMC and tween-80 were added into the flask in different proportions and mixed for a certain period of time.

### 2.3 Dispersion performance evaluation

### 2.3.1 Absorbance evaluation

The absorbance of the dispersion system was measured using an UV-vis spectrophotometer (UV-1700, SHIMADZU Co., China). The prepared dispersion system was allowed to equilibrate for a set amount of time (from 15 min to 135 min), and then, about 1 mL of the suspension was taken using a pipette and placed into a dedicated cuvette to measure its absorbance. The absorbance is directly proportional to the number of the particles per unit volume. Higher absorbance values indicate a better dispersion<sup>[34]</sup>. The absorbance of each sample was measured at the time of 15 min, 45 min, 75 min, 105 min and 135 min. The change of absorbance with time was monitored to evaluate the dispersion stability.

### 2.3.2 FTIR characterization

The chemical structures of modified and unmodified calcium carbonate nanoparticles were analyzed by Fourier transform infrared spectrometer (Nicolet 6700, Thermo Scientific Co., USA). The test samples were ground with potassium bromide (KBr) and analyzed under transport mode from 500 to 4000 cm<sup>-1</sup>.

### 2.3.3 X-ray diffraction characterization

The X-ray diffractometer (DX-2700) was used to characterize the modified and unmodified calcium carbonate nanoparticles. The diffractometer was set up with an electric voltage of 40 kV, a current of 200 mA, a step of 0.02 °/s and a range of  $2\theta = 20^{\circ} - 80^{\circ}$ .

small irregular cracks can be filled by the organic nanomaterials which can deform readily under hightemperature, while the inorganic nanomaterials act as bridging and reinforcing particles due to their rigidity characteristics<sup>[21]</sup>. Calcium carbonate nanoparticles have been widely used because of their low cost, non-toxicity and poor incompatibility between CaCO3 and the polymer matrix<sup>[22-25]</sup>. However, calcium carbonate nanoparticles are mainly produced by a wet chemical precipitation method<sup>[26-27]</sup>, which results in the agglomeration<sup>[28]</sup>. Calcium carbonate nanoparticles have high surface energy and strong surface polarity, easily leading to agglomeration. As a result, the great mass of the calcium carbonate nanoparticles on the market primarily exist as micrometer-sized aggregates/particles. In this way and due to the strong aggregation characteristics, the unique properties and advantages of the nanoscale are lost. Thus, the performance of the calcium carbonate nanoparticles is inferior and their application becomes less appealing<sup>[29-30]</sup>. Avoiding calcium carbonate nanoparticle agglomeration is an important and timely topic for drilling fluid applications as it can enhance its properties. Surface modification is one of the effective methods to solve the dispersion problem of nano powders. Cao et al.<sup>[30]</sup> investigated the effect of surface modifiers on the dispersion of heavy calcium carbonate in the paraffin liquid, and the results showed that isopropyl triisostearate titanate (TTS) could improve the melt fluidity of filler resin and enhance its physical properties. Malti et al.<sup>[31]</sup> studied the calcium carbonate modification by phenylphosphonic acid (PPA) and dodecylphosphonic acid (DPA) in tetrahydrofuran (THF) and ethanol. The results showed that the phosphonic acid monolayer was well coated on the surface of CaCO<sub>3</sub> paticles and the hydrophilicity of CaCO3 was improves. Liu et al.[32] carried out an experimental study on the plugging performance oeveral plugging materials unfer certain temperature and pressure conditions, taking the filtration loss of 30 min as the evaluation standard. The results showed that the ultrafine calcium carte has the best plugging performance and the least filtration loss, which is not abvious affected by temperature on the sealing of microfracture. Wang et al.<sup>[33]</sup> synthesized a new high temperature salt-resistant micrometer and nanometer filter loss reducer. The results of laboratory evaluation experiment showed that the filter loss reducer, with excellent shear dilution property and hightemperature resistance performance, can effectively seal the voids in filter cake, thus reducing the rate of filteration. In this paper, an ultrasonic dispersion technique and dispersants were used to improve the dispersion and resolve the agglomeration of calcium carbonate nanoparticles in aqueous solutions. Calcium carbonate nanoparticles dispersions with enhanced properties were prepared; they showed improved dispersion and better filter loss controlling performance, making them excellent candidates for water-based drilling fluid applications.

### 2. Experimental

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#### 2.3.4 Particle size distribution

A certain amount of calcium carbonate nanoparticles dispersion was stirred evenly, and then, it was added into the sample injector of the laser particle size analysis (MS2000, Malvern analytical Co., UK); the small angle laser light scattering method was used to measure the particle size distribution of the modified and unmodified calcium carbonate nanoparticles.

#### 2.3.5 Thermogravimetric characterization

The thermogravimetric characterization of the modified and unmodified calcium carbonate nanoparticles was performed using a thermal analysis (DSC823e Mettler Toledo Co., Switzerland). Put the sample into the crucible of the thermal analysis at the heating rate of 10 °C·min<sup>-1</sup>. An atmosphere of N<sub>2</sub> was used, and the temperature range covered from 30°C to 800°C.

### 2.3.6 Contact angle evaluation

The modified and unmodified calcium carbonate nanoparticles were pressed into flat sheets. A drop of deionized water was added to the surface of sheet and the wetting angle of water droplet on the calcium carbonate sheet was continuously recorded using an optical contact angle analysis (JY-82C Hebei Chengde Co., China); this allowed the evaluation of the hydrophilicity of the surface of calcium carbonate nanoparticles.

#### 2.3.7 Zeta potential analysis

The multi-frequency acoustic method was used to measure the surface Zeta potential of calcium carbonate nanoparticles. After the calibration of pH probe of the Zeta potential analysis (Zetaprobe, Colloidal Dynamics Co., USA), 250 mL of the suspension was poured into the sample cup and the Zeta potential of the system was measured at the stirring speed of 150 rpm.

### 2.4 The evaluation of depressing filter loss performance

Base mud was prepared with 4 wt.% bentonite, which was stood for 24 h at room temperature. Then, a certain amount of calcium carbonate nanoparticles was slowly added to the bentonite base mud and continued to be stirred for 20 min. The drilling fluid was evaluated according to American Petroleum Institute (API) test programs. At first, the API filtration loss (FL<sub>API</sub>) of the mud was evaluated through the medium pressure mud filtration apparatus (SD3, Qingdao Tongchun Petroleum Instrument Co., China). The smaller the amount of fluid loss is, the better sealing performance as the calcium carbonate nanoparticles block the small pores of shale is. The rheological parameters of the drilling fluid were measured with a six-speed rotational viscometer (ZNN-D6B) at room temperature. Apparent viscosity (AV), plastic viscosity (PV), and yield point (YP) were measured at rotational speeds of 300 rpm and 600 rpm. The formulas used are as follows<sup>[35]</sup>:

$$AV = \frac{1}{2}\Phi 600 \ (mPa \cdot s),$$
  

$$PV = \Phi 600 - \Phi 300 \ (mPa \cdot s),$$
  

$$YP = \frac{1}{2}(\Phi 300 - PV) \ (Pa),$$

Where,  $\Phi 300$  and  $\Phi 600$  represent the numerical value of the rotational speed at 300 and 600 rpm, respectively.

Finally, to further characterize the plugging capacity of calcium carbonate nanoparticles, the microscopic morphology (magnified 20000 times) of the mud cake that formed after the filter loss of base mud was observed by scanning electron microscope (Quanta 450, FEI Co., USA).

### 3. Results and discussion

### 3.1 The optimization and analysis of dispersion conditions

### 3.1.1 The effects of dispersant species and pH value on absorbance

Spectrophotometry was used to analyze the dispersion modification effect of different pH value and dispersants, the concentration of the dispersant was 4 wt.%, the stirring rate was 900 rpm and the dispersion modification time was 45 min. The results are shown in Figure 1.



Figure 1. The effects of dispersant species and pH value on absorbance

Due to drilling fluid being usually weak alkaline, the experiment focused on the dispersion effect of several dispersants at pH values from 7 to 11. It is shown in Figure 1 that the dispersion effect of SDS is the worst and CTAB is the best, and the optimal pH value is 8. The surface of the original calcium carbonate nanoparticles is electronegative and CTAB is a cationic surfactant. CTAB interacts with calcium carbonate particles via van der Waals forces, forming a CTAB absorded layer on the surface of the particles effectively

preventing the agglomeration of calcium carbonate nanoparticles<sup>[36]</sup>. Comparatively, CMC and SDS are anionic surfactants that cannot neutralize the positive charge on the surface of calcium carbonate nanoparticles. As a result, calcium carbonate nanoparticles agglomerate due to the weak repulsion between the particles. Tween-80 is a nonionic surfactant and thus the influence of the pH is diminished. We found that its dispersion effect is similar to CMC.

#### 3.1.2 The effect of stirring time on absorbance

CTAB is selected as the most promising dispersant for optimization. In Figure 2, we present the absorbance curve for the CTAB-calcium carbonate nanoparticles system for different stirring and standing times. The concentration of dispersant is 4 wt.%, the stirring speed is 900 rpm and the pH value is 8.



Figure 2. The effect of dispersion time on absorbance

Figure 2 shows that better dispersion is achieved when the dispersion time is 45 min. The shorter dispersion time is not optimal for the complete adsorption of the dispersant on the calcium carbonate nanoparticles. Conversely, under the sustained shearing action of higher stirring times, the decrease of absorbance of the suspension indicates that the interfacial tension between the solid and liquid is large, and the particles are easy to cluster and sink rapidly. Therefore, the dispersion stability of nano-sized calcium carbonate suspension can be improved by proper modification time.

#### 3.1.3 The effect of stirring speed on absorbance

The effect of stirring speed on the absorbance of calcium carbonate nanoparticles dispersion system is shown in Figure 3 when the concentration of CTAB is 4 wt.%, stirring time is 45 min and the pH value is 8.

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Figure 3. The effect of stirring speed on absorbance

The result indicates that the stirring speed is a significant factor for the dispersion effect of calcium carbonate nanoparticles. When the stirring speed is too slow, the agglomerated particles are difficult to separate. The dispersion is inhomogeneous because of the insufficient interaction between the particles and dispersant. On the other hand, at very high stirring speeds, as the collision between particles intensifies, the interaction between CTAB and CaCO<sub>3</sub> particles is partially destroyed, leading to the formation of new aggregates<sup>[37]</sup>. The optimal stirring speed is 900 rpm.

### 3.1.4 The effect of the concentration of dispersant on absorbance

The change of the absorbance with the concentration of the CTAB dispersant is shown in Figure 4. The stirring speed is 900 rpm, the pH value is 8 and the stirring time is 45 min.



Figure 4. The effect of the concentration of dispersant on absorbance

Figure 4 shows that a CTAB concentration of 4 wt.% achieves the best dispersion effect. When the concentration of the CTAB dispersant is lower, it is insufficient and cannot play

its crucial role. However, when the concentration of the CTAB dispersant is excessive, new aggregates may form due to the irregular contact adsorption, which is undesirable (apart from the fact that using more dispersant is financially costly)<sup>[38]</sup>.

Finally, the optimal process conditions for the dispersion of nano-sized CaCO<sub>3</sub> in water were optimized, that is, the suspension of nano-sized CaCO<sub>3</sub> with pH=8 was ultrasonic for a period of time, then 4 wt.% CTAB was added, and the reaction time was 900 r/min and 45 min at a certain temperature.

3.2 Structure analysis of modified and unmodified calcium carbonate nanoparticles

### 3.2.1 FTIR analysis

FTIR analysis of calcium carbonate nanoparticles before and after modification was performed, and the results is shown in Figure 5.

It can be seen from the figure that nano-CaCO<sub>3</sub> before and after modification both has strong infrared absorption at 1452, 874 and 711 cm<sup>-1</sup>, which is attributed to the stretching vibration and bending vibration of Ca-O. Compared with the unmodified nano-CaCO<sub>3</sub>, the infrared absorption peak of the modified nano-CaCO<sub>3</sub> sample at 3461 cm<sup>-1</sup> becomes stronger, which is greatly related to the stretching vibration absorption of Ca-Br bond. In aqueous solution, CTAB is dissociated into positively charged CTA<sup>+</sup> and negatively charged Br<sup>-</sup>, which form CaBr<sub>2</sub> on the surface of nanoparticles with positively charged calcium ion. In addition, an asymmetrical angular peak of RNH<sup>3+</sup> appears at 1633 cm<sup>-1</sup>, indicating that RNH<sup>3+</sup> has a strong adsorption capacity with nanoparticles after CTAB modification of nano-CaCO<sub>3</sub>, and forms an adsorption layer on the surface of nanoparticles.



Figure 5. FTIR spectra of calcium carbonate nanoparticles before and after modification

### 3.2.2 XRD analysis

The phase changes of calcium carbonate nanoparticles before and after modification were analyzed by X-ray diffraction. The XRD curve is shown in the Figure 6.

As can be seen from the figure 6, both the unmodified and modified calcium carbonate samples show a heavy diffraction peak between  $2\theta = 20^{\circ} \sim 80^{\circ}$ . The diffraction peaks of the modified CaCO<sub>3</sub> are similar to those of the unmodified CaCO<sub>3</sub>, and no new diffraction peaks appear, indicating that the modified CaCO<sub>3</sub> maintains the crystalline phase composition of the original nano-CaCO<sub>3</sub>, and no new crystalline compounds appear.



Figure 6. XRD patterns of calcium carbonate nanoparticles before and after modification

### 3.2.3 Particle size analysis

The particle sizes of the modified and unmodified calcium carbonate nanoparticles in aqueous medium were measured by laser particle size analysis. The particle size distribution is shown in Figure 7. The average particle size( $D_{50}$ ) of the modified and unmodified calcium carbonate particles is 0.593 µm and 2.764 µm, respectively.



### Figure 7. Granularity distribution of calcium carbonate nanoparticles before and after modification

Figure 7 indicates that the serious agglomeration exists in the unmodified calcium carbonate nanoparticles suspension.

According to the particle size distribution from 1  $\mu$ m to 10  $\mu$ m, the dispersion effect is poor. In contrast, the average particle size of the modified calcium carbonate nanoparticles is less than 1  $\mu$ m. Most of particles are approximately distributed in 0.5  $\mu$ m with stable dispersion. However, a small number of particles are still distributed in the range of 1  $\mu$ m to 10  $\mu$ m, which indicates that the agglomeration of nanoparticles still exists to some extent, but the agglomeration degree is weaker than before, which was caused by the static deposition of the particles before the test. Therefore, the stability of the nano-CaCO<sub>3</sub> dispersion system can be maintained by reducing interfacial tension<sup>[39]</sup>.

## 3.3 Performance analysis of modified and unmodified calcium carbonate nanoparticles

### 3.3.1 The thermostability performance analysis of particles

The thermogravimetric performance of nano-sized calcium carbonate before and after modification was measured and the thermogravimetric curve is shown in Figure 8.

As can be seen from the Figure 8, the unmodified CaCO<sub>3</sub> begins to lose weight at about 400 °C. In the range of 400~650 °C, the weight loss rate of the unmodified nano-CaCO<sub>3</sub> is about 1.244%, mainly caused by the reduction of hydroxyl droups on the surface of the particles, while the weight loss rate of the modified nano-CaCO<sub>3</sub> is about 2.384%, maily due to the combustion of CTAB except the reduction of hydroxyl groups on the surface of particles. When the temperature exceeds 650 °C, the nano-meter CaCO<sub>3</sub> begins to undergo intense thermal decomposition, generating solid CaO and CO<sub>2</sub>. Compared with the unmodified calcium carbonate sample, the thermal decomposition rate of the modified calcium carbonate sample is faster, which may be attributed to the interaction of the organic matter and Ca<sup>2+</sup> ions and the introduction of modifier that changes the surface structure of CaCO<sub>3</sub> and promotes the decomposition of nano-meter CaCO<sub>3</sub>.



Figure 8. TG curves of calcium carbonate nanoparticles before and after modification

### 3.3.2 The surface performance analysis of particles

A tablet machine was used to press calcium carbonate nanoparticles into a smooth wafer. Using contact angle analysis, the contact angle was measured by dropping a water droplet on the surface of the wafer. The result is shown in Figure 9.



#### Figure 9. Contact angle test pattern of calcium carbonate nanoparticles (a) before modified (b) after modified

The contact angle of unmodified calcium carbonate nanoparticles is  $25.18^{\circ}$ , while the contact angle of modified calcium carbonate nanoparticles is only  $14.75^{\circ}$ . The result indicates that the dispersants which are absorbed on the surface of calcium carbonate nanoparticles reduce the interfacial tension of the particles, and ultimately promote water to permeate into the interspace of the agglomerated particles. The air in the interspace is exhausted and the agglomeration force is significantly reduced. Finally, the deaggregation of the particles takes place under the action of mechanical force<sup>[40]</sup>.

### 3.3.3 The Zeta potential analysis of particles

To further characterize the dispersion performance, the Zeta potential analyzer was used to analyze the double layer effect of the surface of calcium carbonate nanoparticles. The experimental data is shown in Table 1.

Table 1. Zeta potential of calcium carbonate nanoparticles

-	Sample	Particle concentration	Zeta potential	Conductivity	Dielectric	Kappa value
_		/Wt.%	/mV	/11.5*111	constant	/nm <sup>-1</sup>
	Before modified	2.0	-3.27	0.546	8.5	0.20
	After _' (a)	2.0	34.3	0.4897	8.5	0.29

As shown in Table 1, compared to the unmodified calcium carbonate nanoparticles, the Zeta potential of modified calcium carbonate nanoparticles increases. The Zeta potential was greater than 30 mV, which indicates that the electrostatic repulsion between the particles is more intense and the dispersion system is stable. The modified calcium carbonate nanoparticles can adhere efficiently to the electronegative clay and generate a tight sealing . Thus, it is possible to apply the modified calcium carbonate nanoparticles as filter loss additives in drilling fluids.

### 3.4 Filter loss controlling performance evaluation

The filter loss performance and rheological property of drilling fluid were tested by filter testing and rotational viscometry, respectively. The properties of the base mud, mud containing modified or unmodified calcium carbonate nanoparticles are shown in Table 2.

 Table 2 Performance measurements of the drilling mud with

 calcium carbonate nanoparticles

Formulation	AV/ mPa·s	PV∕ mPa∙s	YP/ Pa	FL <sub>API</sub> / mL
Base mud	21.0	9.0	12.0	22
Base mud+1 wt.% unmodified CaCO3 nanoparticles	15.5	6.0	9.5	13.5
Base mud+1 wt.% modified CaCO3 nanoparticles	13.2	6.0	7.2	6.5

As shown in Table 2, after adding the modified or unmodified calcium carbonate nanoparticles in the drilling fluid, the filter loss (FL<sub>API</sub>) is less than the base mud. The filter loss of the drilling fluid added with modified calcium carbonate nanoparticles reduces more significantly in comparison, which indicates that more of the narrower pores are plugged by the modified calcium carbonate nanoparticles and more compact mud cake is formed. The apparent viscosity (AV), plastic viscosity (PV) and dynamic shear force (YP) show a decreasing trend. The rheological properties of the drilling fluid are improved, which is favorable for the pumping capability of the drilling fluid.

The micro-morphology images of the mud cake formed by the modified and unmodified calcium carbonate nanoparticles were acquired by scanning electron microscope at 20,000 times magnification. The images are shown in Figure 10.



Figure 10. Scanning electron micrographs of the mud cake formed by (a) unmodified calcium carbonate nanoparticles and (b) modified calcium carbonate nanoparticles

As shown in Figure 10, the mud cake of the drilling fluid added with unmodified calcium carbonate nanoparticles is rougher and contains pores; the aggregation of particles prevents them from entering into the smaller pores (Figure 10 (a)). However, the mud cake formed by the drilling fluid added with modified calcium carbonate nanoparticles is more compact, smoother and the particles are more evenly dispersed (Figure 10 (b)). The modified calcium carbonate nanoparticles plugged the tiny pores of the mud cake and achieved a better plugging effect.

### 4. Conclusions

The dispersion effect of calcium carbonate nanoparticles was investigated by spectrophotometry. Optimal dispersion effect was achieved at a CTAB concentration of 4 wt.%, stirring speed at 900 rpm, modification time of 45 min and pH value of 8. Under these optimal conditions, the absorbance of the dispersion system reached its maximum and the hydrophilicity and the surface double layer of the calcium carbonate nanoparticles was improved after dispersion modification.

FTIR and XRD analysis showed that the surface structure of nano-CaCO<sub>3</sub> was changed after modification, and the chemical adsorption layer, which had no effect on the crystal structure of nano-CaCO<sub>3</sub>, was formed on the surface of particles by CTAB. The Contact Angle test showed that the surfactant adsorbed on the surface of nano-CaCO<sub>3</sub> particles can reduce the interfacial tension of particles and the agglomeration force between particles effectively. When the particle size of nano-CaCO<sub>3</sub> is large, the micro-sized and nano-sized pores cannot be effectively blocked, reducing drilling efficiency. Thus, the average particle size ( $D_{50}$ ) of the modified calcium carbonate nanoparticles decreased from

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58 59 60  $2.764 \ \mu m$  to  $0.593 \ \mu m$ , which indicates that the agglomeration of the particles was effectively restrained. The mud cake formed by base mud added with modified calcium carbonate nanoparticles is more compact and the tiny particles are dispersed more evenly. The API filter loss also decreased significantly and the plugging performance was improved.

### 5. Acknowledgments

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