Performance of carbonate calcium nanoparticles as filtration loss control agent of water-based drilling fluid

Farshad Dehghani^{1,5} · Azim Kalantariasl^{1,2} · Rahmatallah Saboori³ · Samad Sabbaghi⁴ · Kiana Peyvandi⁵

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Abstract

Application of nanoparticles in improvement of drilling fluid properties through effective control of fluid loss and rapid formation of thin, smooth and low permeable filter cake has gained considerable attention in recent years. In this study, calcium carbonate nanoparticle was synthesized by precipitation method and characterized by X-ray diffraction, Fourier transform infrared spectroscopy, scanning electron microscope (SEM), dynamic light scattering and Zeta-potential measurement. Water based bentonite drilling fluid was used as base fluid. Various concentrations of calcium carbonate nanoparticle (0.025–0.5 wt%) was added to water-based drilling fluid. Fluid loss volume, filter cake thickness and its surface morphology, and rheological properties of both base fluid and fluid containing calcium carbonate nanoparticles was measured and compared. Results show that addition of calcium carbonate nanoparticle greatly affects filtration properties and forms smoother cake surface. Optimum concentration of 0.07 wt% calcium carbonate nanoparticle was obtained which results in reduction of fluid loss volume and filter cake thickness by 26% and 64% respectively while minor change in rheological behavior was observed. SEM image showed smoother cake surface of drilling fluid with calcium carbonate nanoparticle as additive compared to base drilling fluid. Considerable improvement in filtration properties using acid soluble CaCO_3 nanoparticles can help in minimizing fluid leak-off and formation damage of producing layers and more effective well cleanup for fast oil and gas production.

Keywords Nanoparticle · Calcium carbonate · Drilling fluid · Fluid loss · Mud cake · Rheological properties

1 Introduction

Drilling fluid is a vital part of oil, gas and geothermal drilling operations. Drilling fluid consists of a base fluid (water/oil), solid additives and chemicals. Many additives are used in drilling fluid since it has many functions including supporting formation pressure, cleaning of the wellbore, cutting transport and removal, cooling tubular, minimizing formation damage through rapid buildup of external filter cake and preventing fluid loss into the subsurface formations, etc. It is widely accepted that almost all drilling problems are directly or indirectly related to drilling fluid. Thus

proper drilling fluid design is a key issue in safe and cost effective drilling operations [1–3].

Among drilling fluid functions, preventing fluid loss into the subsurface formations is very important for formation damage control, wellbore stability, and loss of fluids into the formation which can be optimized by selection of proper additives. Along filtration control, formation of thin low permeable filter cake on the well wall has critical role in reduction of fluid loss and strengthen the wellbore by minimizing contact between drilling fluid and adjacent formation through reduction of fluid loss [3–6].

✉ Azim Kalantariasl, Azim.kalantari@shirazu.ac.ir | ¹Department of Petroleum Engineering, School of Chemical and Petroleum Engineering, Shiraz University, Shiraz, Iran. ²Formation Damage and Well Treatment Research Group, IOR/EOR Research Institute, Shiraz University, Shiraz, Iran. ³Department of Chemical Engineering, Lar University, Lar, Iran. ⁴Department of Nano Chemical Engineering, School of Advanced Technologies, Shiraz University, Shiraz, Iran. ⁵School of Chemical Engineering, Semnan University, Semnan, Iran.

Nanoparticles have been proposed for wettability alteration, asphaltene deposition mitigation and removal, drilling fluid performance enhancement [7–10]. In drilling operations, they have been used for shale inhibition, fluid loss control, thermal conductivity improvement, enhancement of rheological properties [9, 11–13].

Many nanoparticles are investigated to improve drilling fluid properties in the literature. Silica nanoparticles have been employed to decrease water absorption into shale formation and reducing drilling problems such as pipe sticking, shale hydration and wellbore stability [6, 14–16]. CMC nanoparticles have been applied for rheology improvement, filter cake thickness reduction and filtration control [9, 17]. Fe_3O_4 has been used for improvement of drilling fluid properties at high pressure high temperature (HPHT) conditions [18].

Invasion of drilling fluid solids and filtration of water into hydrocarbon bearing formation can result in many problems [19]. Water can change near wellbore wettability which affects oil production. Different chemistry of water may detach fine particles from grain surface and alter permeability which is a severe problem that can defer production and may not be cured in some cases. Thus minimizing formation damage is a desirable issue that can be achieved with proper mud design and additive selection [2–4].

In this study, the effect of calcium carbonate nanoparticle on filtration and rheological properties of water-based drilling fluid was investigated. Calcium carbonate nanoparticle was chosen since it is a cost-effective acid soluble particle that can be used in hydrocarbon pay zones. Calcium carbonate nanoparticle was synthesized and characterized. Then nanoparticle was added to drilling fluid and filtration and rheological properties were measured. The effect of calcium carbonate nanoparticle concentration on fluid loss, cake thickness and surface morphology, and rheological properties was investigated and optimum concentration with minimum fluid loss and cake thickness was obtained.

2 Experimental section

2.1 Materials

Sodium carbonate, calcium acetate, polyethylene glycol (PEG), and ethanol (> 98%) were purchased from Merck company. Deionized water was perched from Zolal Company. Bentonite was supplied from National Iranian Drilling Company.

2.2 Synthesis of calcium carbonate nanoparticle

In this study, calcium carbonate nanoparticles were synthesized by chemical precipitation method. To synthesize nanoparticle, 5 g of sodium carbonate and polyethylene glycol were added to 20 ml of deionized water and stirred with a magnetic stirrer (solution A). Then 3 g of calcium acetate was added to 25 ml of deionized water and the solution stirred for 40 min by magnetic stirrer (solution B). Solution B was added to solution A drop wise while stirring with vigorous stirring at ambient conditions. The process was completed after 2.5–3 h at ambient condition. Then, the product was allowed to precipitate completely. The sediment dried and then the final powder was placed in 100 °C. The synthesized calcium carbonate nanoparticle was characterized using X-ray diffraction (XRD) and dynamic light scattering (DLS). The composition blend of sample was examined via a Unisantix XMD 300, X-ray diffract meter and systematic Xpert PRO X-ray diffraction ($\lambda = 0.17890$ nm). 2θ confine used was starting 30–75 in stages of 0.02 by enumerate time of 1 s. The Fourier transform infrared spectroscopy (FTIR) was examined via SHIMADZU 8300 FTIR spectroscopy for bonds of synthesized calcium carbonate nanoparticle. TESCAN VEGA3 scanning electron microscope (SEM) was used to determine the morphology of synthesized nanoparticle.

In order to prepare calcium carbonate nanoparticles dispersion for zeta-potential and particle size distribution measurement, nanoparticles were gradually added to deionized water and mixed by vigorous magnetic stirrer for 30 min at 25 °C. Then, the suspension was sonicated by ultrasonic device with high power (20 kHz and 400 W) for 30 min at 25 °C using water bath for temperature control. It is noteworthy that no dispersant and stabilizer were used for dispersion of nanoparticles in base fluid. Size distribution of calcium carbonate nanoparticles was recorded by a Horiba-LB-550 dynamic light scattering (DLS). DLS equipment measures particle size in the rage of 1 nm to 6 μm . To evaluate the stability of calcium carbonate nanoparticles to be used in water-based drilling fluid, zeta-potential of nanofluid was measured by Microtrac zeta-check.

2.3 Preparation of drilling fluid

To prepare base drilling fluid, 10 g of bentonite powder was gradually added to 350 ml of deionized water and mixed by Hamilton Beach mixer with 36,000 rpm for 20 min.

To prepare Nano drilling fluid, calcium carbonate nanoparticle with different concentrations was added to

100 ml deionized water and mixed with magnetic stirrer for 30 min and then, sonicated with 20 kHz and 300 W for 15 min (solution A). This process was performed to monitor stability of nanoparticles (calcium carbonate) before addition to base drilling. In addition, 10 g of bentonite, and 250 ml of deionized water were mixed by Hamilton Beach Mixer for 10 min with 36,000 rpm (solution B). The solution A was dropwise added to solution B and mixed by Hamilton Beach Mixer for 15 min.

Filtration properties such as fluid loss and filter cake thickness of drilling fluid were measured using filter press device according to API standard. Filter press at 100 psi and 25 °C was used for monitoring fluid loss versus time. Fluid loss was recorded every 5 min to compare the effect of calcium carbonate nanoparticle concentration on cumulative fluid loss. The required time to measure the fluid loss and filter cake thickness is 30 min as industry standard. Then optimum calcium carbonate nanoparticle was obtained. SEM of cake surface on the filter paper was taken for both base drilling fluid and drilling fluid with optimum concentration samples to compare the effects of calcium carbonate additive on cake surface. Rheology test (shear stress vs. shear rate) for base fluid and fluid with optimum concentration were also performed.

3 Results

3.1 Characterization of calcium carbonate nanoparticle

The XRD pattern of synthesized calcium carbonate nanoparticle is shown Fig. 1. The XRD analysis demonstrates the characteristic peaks at 26.5, 27.7, 33.4, 45.9 and 51.5° corresponding to the calcium carbonate. The peaks are in good agreement with the crystalline planes of calcium

carbonate nanoparticle reported in the literature [20–22]. The crystal size of the synthesized calcium carbonate nanoparticle was estimated about 40 nm by using the Debye–Scherrer formula.

Figure 2 shows the FTIR spectrum of synthesized calcium carbonate nanoparticle by precipitation method. The bands at 712, 848 and 872/cm were attributed to the CO_3^{2-} (calcite) vibration mode of calcium carbonate nanoparticle. The symmetric stretching vibrational of CO_3^{2-} and C–H band was observed at 1406 and 2850/cm, respectively. Also, the bands were detected at 2513 and 2916/cm, which indicated the calcite and C–C (mode of vibration). The bond at 1795 was assigned to the calcite vibration mode [21, 23–26].

Figure 3 shows the morphology of calcium carbonate nanoparticles. The synthesized CaCO_3 consists of semi-spherical particles and the particle size is < 60 nm.

Fig. 2 FTIR analysis of synthesized calcium carbonate nanoparticle

Fig. 1 The XRD pattern of synthesized calcium carbonate nanoparticle by precipitation method

Fig. 3 FE-SEM image of calcium carbonate nanoparticle

Fig. 4 Particle size distribution (DLS) of calcium carbonate nanoparticle

Figure 4 shows the particle size distribution (DLS) of synthesized calcium carbonate nanoparticle. Average particle size of nanoparticle is 55.4 nm and particle size distribution is narrow in water based fluid.

Table 1 shows absolute values of zeta-potential for different concentrations of calcium carbonate nanofluid in water base fluid. Nanofluids with all concentrations less than 0.15 wt% had excellent stability (> 60 mV) which confirm the stability conditions.

Table 1 Zeta-potential of calcium carbonate nanofluid at different concentrations

Nanofluid concentration (wt%)	Absolute zeta-potential (mV)
0.025	73.0
0.050	68.0
0.070	66.3
0.150	63.8
0.500	55.5

Fig. 5 Effect of nano CaCO_3 concentration on fluid loss (30 min)

3.2 Characterization of drilling fluid

3.2.1 Drilling fluid filtration test

The effect of calcium carbonate nanoparticle concentration on fluid loss is shown in Fig. 5. Filtration for 30 min at 100 psi pressure and measuring fluid loss volume via filter press device which is standard in drilling industry is presented for different calcium carbonate nanoparticle concentrations. Increase in nanoparticle concentration from 0.025–0.07 wt% causes sharp decrease in fluid loss from 27 to 20 cc. from 0.07 to 0.5%, the change in fluid loss is not significant. It shows 26% decrease in fluid loss volume just by addition of 0.07% calcium carbonate nanoparticle which is considerable in large volume of drilling fluid used daily in oil and gas drilling operations.

Figure 6 shows the effect of calcium carbonate nanoparticle concentration on cumulative fluid loss (total fluid loss during 30 min filtration) that has been recorded every 5 min.

Regarding calcium carbonate nanoparticle concentration effect, highest cumulative fluid loss corresponds to 0.025 wt% while for 0.07, 0.15 and 0.5 wt% the cumulative fluid loss are almost the same which confirms

Fig. 6 Cumulative fluid loss in the presence of different concentration of calcium carbonate nanoparticle versus time

Fig. 8 Rate of fluid loss in the presence of different concentration of calcium carbonate nanoparticle for whole time span

Fig. 7 Rate of fluid loss in the presence of different concentration of nano CaCO_3

good performance of calcium carbonate nanoparticle at desired concentrations.

Initial rate of fluid loss (during 5 min interval) for base mud (without calcium carbonate nanoparticle), 0.025 and 0.05 wt% calcium carbonate nanoparticle are 2, 2 and 1.7 cc/min respectively while for 0.07, 0.15, and 0.5 wt% is 1.6 cc/min which shows 20% decrease in rate of fluid loss at initial filtration time. Less fluid loss rate corresponds to better cake formation which is desired in drilling operations.

Figure 7 presents rate of fluid loss (derivative of cumulative fluid loss vs. time, Fig. 6) as a function of time for base fluid and different calcium carbonate nanoparticle concentrations.

In order to better demonstrate the effect of nano CaCO_3 concentration on rate of fluid loss, Fig. 8 shows fluid rate in whole time span (derivative of cumulative fluid loss vs. time but not local time as presented in Fig. 6) and the effect of calcium carbonate nanoparticle concentration on rate of fluid loss is significant both for initial fluid loss rate and rate versus time.

Fig. 9 Thickness of mud cake in the presence of nano CaCO_3 in drilling fluid

It should be mentioned that, all experiments were repeated three times and the average of results is reported here.

3.2.2 Filter cake properties

Cake thickness Cake thickness plays an important role in drilling operations of oil, gas and geothermal wells. Formation of thin and low permeable external filter cake on the wall of the formation is crucial for minimization of fluid loss into the adjacent formation, strengthen and stability of the borehole, and reducing risk of pipe sticking. Figure 9 shows filter cake thickness obtained for different concentrations of calcium carbonate nanoparticle added to the base drilling fluid. It shows that addition of small amount of calcium carbonate nanoparticle significantly reduced cake thickness (from 2.8 mm to almost 1 mm). It seems that on average and considering fluid loss results (Fig. 5), an optimum concentration of 0.07 wt% calcium carbonate

nanoparticle results in 26% decrease in fluid loss and 64% decrease in cake thickness which is a great achievement both from economical and operational aspects. Thus, optimum conditions can be achieved considering both fluid loss volume and cake thickness during 30 min filtration.

Cake surface The morphology of mud cake surface after filtration test and drying is shown in Fig. 10. SEM Image of base drilling fluid (without carbonate calcium nanoparticle) is presented in Fig. 10a. Figure 10b shows SEM image of cake surface using 0.07 wt% calcium carbonate nanoparticle. It shows more homogenous and smoother surface compared to cake surface of base drilling fluid.

Fig. 10 SEM image of **a** water-based drilling fluid without nanoparticle, **b** water-based drilling fluid with 0.07 wt% of calcium carbonate nanoparticle

Addition of nanoparticle can improve cake formation quality with filling small pores and making smoother cake surface. It results in less fluid filtration and subsequent thinner cake formation which both are desirable drilling fluid properties for drilling operations. It is favorable to form a low permeable cake at early time of filtration since it reduces fluid loss and consequently thin cake can be formed in dynamic drilling fluid filtration.

3.2.3 Rheological test

Rheological behavior of 0.07 wt% calcium carbonate nanoparticle concentration and base drilling fluid is presented in Fig. 11. It shows Bingham plastic model for both fluids which is typical for drilling fluid. One can see that despite improvement in filtration and cake thickness, rheological behavior does not change significantly that may be due to low concentration of calcium carbonate nanoparticle needed for such an improvement. It is noteworthy that, the calcium carbonate nanoparticles used in this study have no effect on the mud density and pH of drilling fluid for all concentrations.

4 Discussion

Filtrate-loss volumes are used to compare the performance of various fluid types in drilling industry. The proposed nano-particle in this paper shows promising results to be used in water-based drilling fluid as fluid loss control. Reduction of fluid filtration into underground formations greatly affects economic of drilling operations. Moreover small cake thickness is desirable in drilling operations since it greatly reduces pipe sticking and consequently non-operating time of drilling rig. This is very important in drilling of horizontal wells where pipe sticking is more likely to happen frequently.

Fig. 11 Rheological behavior of 0.07 wt% calcium carbonate nanoparticle and base drilling fluid

The performance of nano CaCO_3 can be compared with existing filtration control agents. Filtration control is critical for drilling pay zones (formations containing oil/gas) to minimize fluid leak-off and formation damage. It is very common to use acid soluble CaCO_3 particles as fluid loss control additive when drilling oil and gas producing formations since after drilling it is required to remove filter cake from the wall of the well bore (well clean up) for oil and gas production. Thus using nano- CaCO_3 in drilling reservoir sections is superior to other agents due to promising performance in filtration (less filtrate, thin and smoother cake) and its solubility in HCl.

Static and dynamic filtration of proposed nano-particle using core plugs from target formation in core-flooding system can be used to evaluate its performance in desired pressure and temperature conditions [3]. Moreover, using real core sample from different formations allows obtaining filtration performance of proposed nano particle in more realistic conditions.

Elemental analysis of filter cake surface can improve our understanding of performance of additives. Visual observation of cake surface and layers using micro computed-tomography (CT) scanning can give insights about mechanisms behind performance of nano CaCO_3 in drilling fluid [27, 28].

5 Conclusions

Carbonate calcium nanoparticle was synthesized via precipitation method and characterized by XRD, FTIR, SEM, DLS and zeta-potential measurement and the synthesized nanoparticles were used as filtration reducer in water-based drilling fluid.

- Fluid loss was considerably reduced with addition of carbonate calcium nanoparticles.
- Optimum concentration of carbonate calcium nanoparticle (0.07 wt%) results in 26% decrease in fluid loss and 64% reduction of cake thickness.
- SEM image of cake surface confirmed smoother cake surface when using carbonate calcium nanoparticle as drilling fluid additive.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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