



# Improvement in filtration properties of water-based drilling fluid by nanocarboxymethyl cellulose/polystyrene core–shell nanocomposite

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## Abstract

Since almost all drilling problems directly or indirectly relate to drilling fluid, improvement in drilling fluid significantly enhances drilling operations. Drilling fluid contains base fluid, suspended solid particles and chemicals. Recently, nanoparticles have been widely recommended for improvement in drilling fluid properties. The main purpose of this study was to improve the rheological and filtration properties of water-based drilling fluid through adding new additive carboxyl methyl cellulose (CMC)/polystyrene core–shell nanocomposite. It compares filtration and rheological behavior of CMC, nanoCMC and core–shell nanocomposite. The core–shell nanocomposite was synthesized by miniemulsion polymerization method, and nanoCMC and core–shell nanocomposite were characterized by scanning electron microscope, transmission electron microscopy, particle size analyzer and thermogravimetric analysis. Fluid loss, mud cake thickness, viscosity, weight and pH of drilling fluid with core–shell nanocomposite additive were compared with conventional CMC (bulk CMC) and nanoCMC particles. Results showed a significant decrease in mud cake thickness and fluid loss when the core–shell nanocomposite was used, as compared with conventional CMC and nanoCMC. Viscosity of three additives has same trend with insignificant change while less yield point is obtained for drilling fluid containing core–shell nanocomposites. Mud weight and pH were almost the same for all three additives. Thus, the core–shell nanocomposite can be an alternative additive to control mud cake thickness and fluid loss while maintaining other main properties in an acceptable range.

**Keywords** Drilling fluid · Core–shell nanocomposite · CMC nanoparticle · Polystyrene · Filtration · Rheology

## Introduction

Drilling fluid has many key functions in drilling operations, and almost all problems encountered in drilling operations are directly or indirectly related to drilling fluid properties (Adams 1985; Chilingarian and Vorabutr 1983; Patel 1998; Plank and Gossen 1991). Optimum selection of drilling fluid is a key factor in minimizing drilling time and cost

(Mokhtari and Ozbayoglu 2010; Salih et al. 2016). Due to cost, environmental issues, water-based drilling fluid is more preferable and attractive option than oil and synthetic fluids for drilling oil and gas wells in sensitive areas where oil base fluids are not desired. Development of high performance and more environmental friendly water base fluids are desirable (Salih et al. 2016).

Water-based drilling fluid mainly consists of water as base fluid, inert and reactive solids as additives which still has many disadvantages including shale instability, formation damage, poor cake properties and high fluid loss; recently, nanowater-based fluid has been proposed by several researchers for overcoming related issues in drilling fluid such as reducing torque and drag, controlling fluid loss, minimizing formation damage, improving wellbore stability and subsequently improving drilling performance. Due to high surface to volume ratio of nanosized particles, they can change chemical and physical properties of drilling fluids with low concentration. Several studies have proven

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superiority of NBFs to conventional drilling fluids (Garcia-Ochoa et al. 2000; Li et al. 2015a; Pérez et al. 2004; Singh et al. 2010; Tabzar et al. 2015; Yu 2015; Wang et al. 2016).

Wang et al. (2011b) investigated the effect of cross-linked polystyrene microsphere as a density-reducing agent on drilling fluid.

William et al. (2014) studied the effects of CuO and ZnO nanofluids on thermal, electrical and rheological properties of the water-based drilling fluid at different pressures. Their results showed that addition of nanoparticles increases thermal and electrical properties of drilling fluid by about 35%. In addition, increasing nanoparticle concentration caused increase in the thermal and electrical properties. Sadeghalvaad and Sabbaghi (2015) reported decrease in filtration and increase in mud viscosity when TiO<sub>2</sub>/polyacrylamide nanocomposite used as additive to water-based drilling fluid.

Sensoy et al. (2009) observed reduction in absorbed water by shale from 16 to 72% when silica nanoparticle was used. Ragab and Noah (2014) showed that adding silica nanoparticles in drilling fluid caused a decrease in formation damage, fluid loss and consequently improvement in drilling operation. Wang et al. (2011a) reported the application of modified polyacrylamide nanoparticles in separation of solid and liquid phases in the wastewater of drilling fluid. The results represented decrease of 33–56% in fluid loss. Zoveidavianpoor and Samsuri (2016) investigated the effects of nanosized tapioca starch nanoparticles for filtration control in water-based drilling fluid and showed improvement in fluid loss by 64.2% and reduction in the mud cake thickness by 80.9%. Barati (2015) studied the effects of silica and polyelectrolyte complex nanoparticles as fluid loss control additives for hydraulic fracturing of tight and ultra-tight core plugs and reported reduction in filtration. Saboori et al. (2012) observed intensive reduction in the fluid loss and mud cake thickness by using CMC nanoparticle (nanoCMC) compared to bulk CMC. Improvement in the sealing ability, reduction in mud cake permeability and wellbore stability was observed by adding nanoparticles compared to calcium carbonate as reported by Li et al. (2012). Halali et al. (2016) used CNT nanoparticle as an additive to drilling fluid for increasing thermal stability of polymeric fluid. The results showed reduction in filtration by 95% at high-temperature conditions and increase in stability and zeta potential of the shale. Kosynkin et al. (2011) used the graphene oxide nanoparticle and showed reduction in filtration rate compared to clays and polymers standard suspension. Xuan et al. (2014) investigated the graphite oxide nanoparticles and showed it has a strong ability in controlling fluid loss of drilling fluid. Li et al. (2015b) used cellulose nanoparticles to improve the rheological properties and fluid loss in water-based drilling fluid and reported. Benyounes et al. (2010) investigated the effect

of anionic polymer such as carboxymethyl cellulose and xanthan on rheology of bentonite suspension at different concentrations. Results show that carboxymethyl cellulose helped to remove yield stress and increase viscosity but xanthan caused increase in yield stress and high increase in viscosity of the bentonite–polymer mixtures. Kalantariasl et al. (2014) studied effect of particle size, size distribution and applied pressure on cake formation mechanisms during dynamic filtration of drilling fluids and poor quality water injection process.

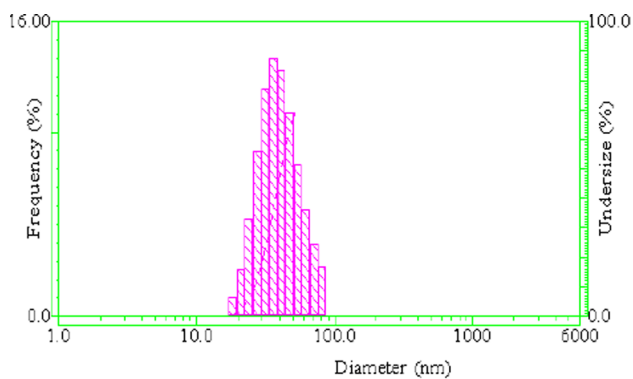
However, selection of type, concentration and size of nanomaterial additives that best fit the conditions for different purpose in drilling operations is still under investigation and needs deeper knowledge for different conditions. This study investigates and compares rheological and filtration performance of CMC (as common additive in conventional drilling fluid), nanoCMC and core–shell nanocomposite. All materials are synthesized and characterized with several tools. Then, synthesized materials are added to water-based drilling fluid to investigate possible improvement in properties and performance with standard methods. In this study, core–shell nanocomposite of nanoCMC with polystyrene by miniemulsion polymerization was synthesized and characterized by PSA, SEM, TEM and TGA analyzer. Then, the synthesized core–shell nanocomposite was added to the conventional water-based drilling fluid according to the API standard (API 2003). Finally, main drilling fluid properties such as mud cake thickness, fluid loss, plastic viscosity, apparent viscosity, weight (density) and pH were monitored. The results are compared with nanoCMC and bulk CMC.

The structure of the paper is as follows: experiments, materials and methods used for synthesis of nanocomposite and preparation of drilling fluids are described in second section. Results are presented in third section. Discussion of results and conclusions is presented in third and fourth sections, respectively.

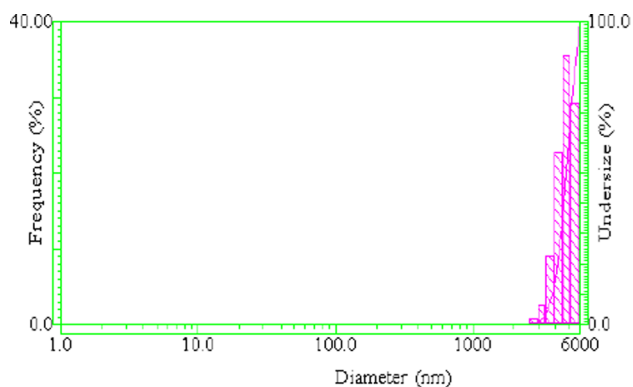
## Experiments and procedures

### Materials

In this section, materials and methods for preparation of nanocarboxymethyl cellulose/polystyrene core–shell nanocomposite is explained. NanoCMC was purchased from Nano Technology Research Institute (Shiraz University). The average particle size of nanoCMC (about 47 nm) measured using particle size analyzer (PSA) was used to measure particle size distribution (see Fig. 1). Average particle size of nanoCMC was about 47 nm. Hydrochinon, benzoyl peroxide, sodium dodecyl sulfate (SDS) as surfactant, styrene with



**Fig. 1** Particle size distribution of CMC nanoparticle (nanoCMC)



**Fig. 2** Particle size distribution of bulk CMC

a 20-ppm inhibitor (analytical grade), span80 (surfactant) and hexadecane were purchased from Merck Co. Bentonite and bulk CMC were purchased from National Iranian Oil Company (NIOC). The average particle size of bulk CMC was measured using particle size analyzer (6  $\mu\text{m}$ ) (see

Fig. 2). Comparison of particle size distribution (Figs. 1, 2) shows significant difference between bulk and nanoCMC.

### Synthesis of core–shell nanocomposite

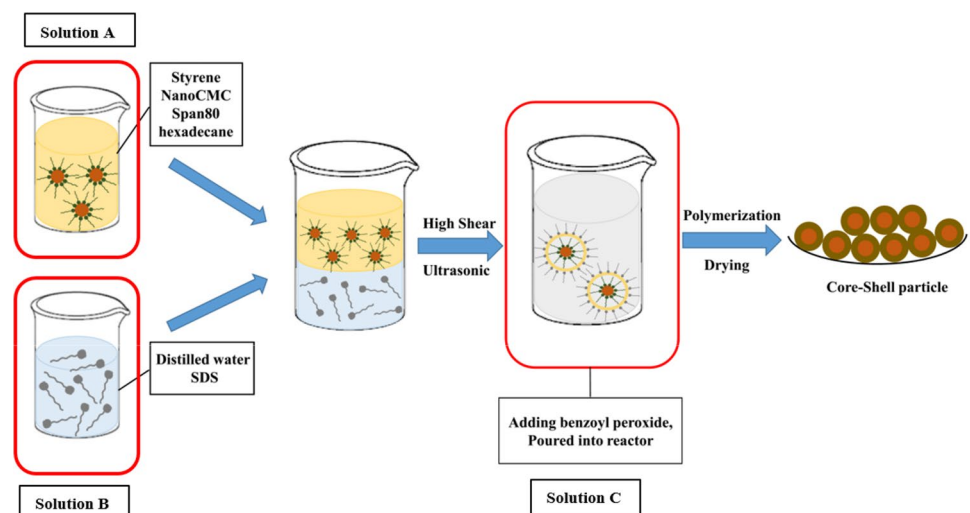
The process used to prepare the core–shell nanocomposite is as follows (see Fig. 3): nanoCMC (0.6 g), styrene (25 cc), hexadecane (1.5 g) and span80 (0.7 g) were added in a beaker and mixed by vigorous magnetic stirrer for 30 min and then sonicated using the ultrasonic device (solution A) for 15 min. A solution of sodium dodecyl sulfate (SDS) (0.45 g) and distilled water (70 cc) was mixed in another beaker by magnetic stirrer for 10 min (solution B). Then, the abovementioned mixtures were sonicated by the ultrasonic irradiation for 20 min (solution C) as shown in Fig. 3.

Solution C and benzoyl peroxide (0.5 g) were poured into a 500-mL round-bottomed reactor under the nitrogen and condenser with a mechanical mixer fixed at 300 rpm during the process (see Figs. 3, 4). The fixed temperature (70  $^{\circ}\text{C}$ ) was controlled by water bath, and mechanical stirring rate (300 rpm) was constant during the whole process (Fig. 4). Sampling was carried out every 30 min along the polymerization process. Each sample (2 cc) was inhibited by using the one drop of hydroquinone (1 wt%)/methanol solution. Eventually, the samples were dried at room temperature and used to calculate the conversion percentage, i.e., the progress of polymerization was monitored every 30-min sampling. The whole process was completed after 8–9 h. Finally, the product (core–shell nanocomposite) was cooled at room temperature and dried on 80–100  $^{\circ}\text{C}$

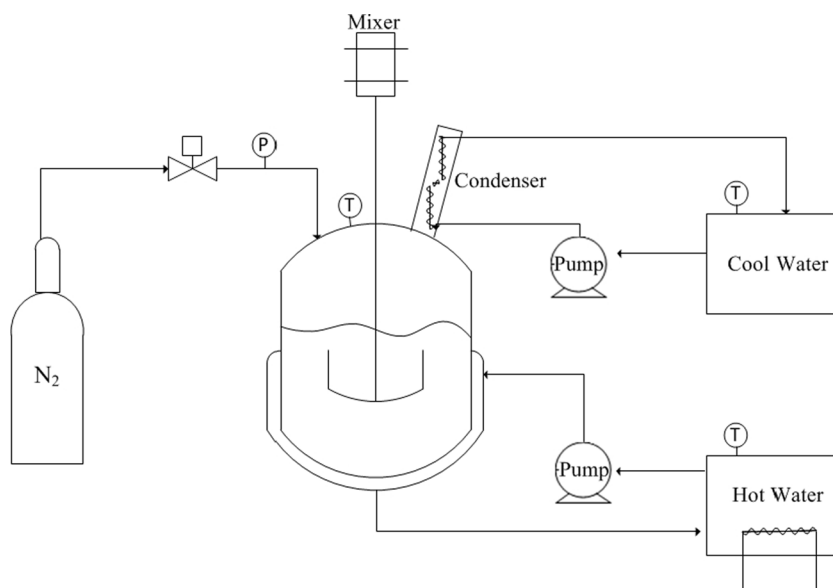
### Core–shell nanocomposite characterization

This section presents characterization of core–shell nanocomposite as described in the previous section. Scanning electron microscope (SEM), transmission electron

**Fig. 3** Schematic of polymerization process for synthesis of core–shell nanocomposite



**Fig. 4** The schematic of the experimental setup used for polymerization process



microscopy (TEM), particle size analyzer (PSA) and thermogravimetric analysis (TGA) were used to characterize the core–shell nanocomposite and nanoCMC. SEM and TEM are digital systems that can help in visualization and interpretation of morphology, size distribution and verification of the core–shell nanocomposite. The TGA analysis determines the thermal resistance of the synthesized core–shell nanocomposite. The micrograph of the core–shell nanocomposite was analyzed by SEM (Fig. 5a) and TEM (Fig. 5b). SEM micrograph shows that the core–shell nanocomposites have almost spherical shape (Fig. 5a). TEM analysis indicates dark CMC nanoparticle (nanoCMC) cores (about 45 nm) and light polystyrene shells (about 30 nm) around CMC nanoparticles and core–shell nanocomposite have almost spherical shape (Fig. 5b).

Figure 6 shows particle size distribution of core–shell nanocomposite with almost normal distribution and average size of 80 nm.

Thermal analysis (TGA) of core–shell nanocomposite and nanoCMC was carried out with a Perkin Elmer Pyris thermal analyzer. The results of TGA curves are shown in Fig. 7, which indicates that the core–shell nanocomposites are much more thermally stable than nanoCMC. Hence, it is concluded from the TGA results that the graft of polystyrene chains on to the polysaccharide backbone enhances the thermal stability of the polysaccharides. Thus, the nanoCMC/polystyrene core–shell nanocomposite has higher thermal stability. Compared to nanoCMC, core–shell nanocomposites are stable in a wide range of temperatures, which is important for high-temperature conditions (i.e., drilling high-temperature formations in geothermal and oil/gas fields).

### Preparation of nanowater-based drilling fluid

In order to prepare water-based drilling fluid, distilled water (350 mL) and bentonite (10 g) were mixed by Hamilton batch mixer (36,000 rpm) for 20 min at room temperature according to API standard (API 2003; Api 2014). The synthesized core–shell nanocomposites, nanoCMC and bulk CMC were separately (1–10 g) added to the drilling fluid and were stirred by the Hamilton batch mixer for 15 min. Prepared drilling fluid samples (with core–shell nanocomposite, nanoCMC and bulk CMC as additives) were used to measure and compare fluid loss, mud cake thickness, viscosity, mud weight and pH.

### Measurement of drilling fluid properties

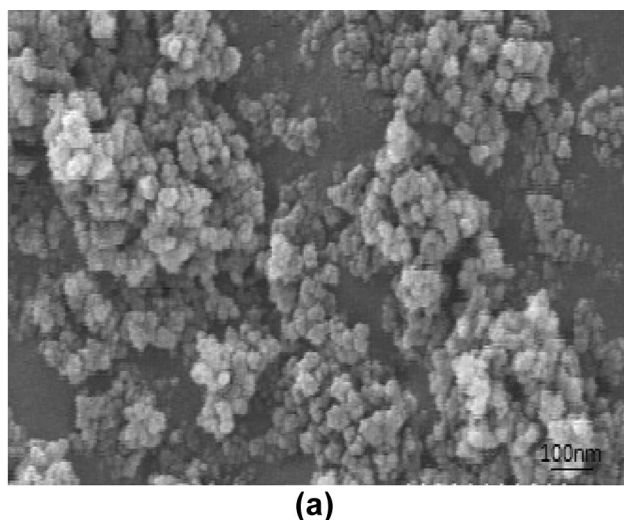
**Viscosity** The viscosity of three mentioned drilling fluid samples were measured using Fann 35 at the speeds of 300 and 600 rpm, room temperature and atmospheric pressure. Afterward, apparent viscosity (AV), plastic viscosity (PV) and yield point (YP) were calculated using the following equations (Bourgoyne et al. 1991).

$$AV = \frac{1}{2}\theta_{600} \quad (\text{cP}) \quad (1)$$

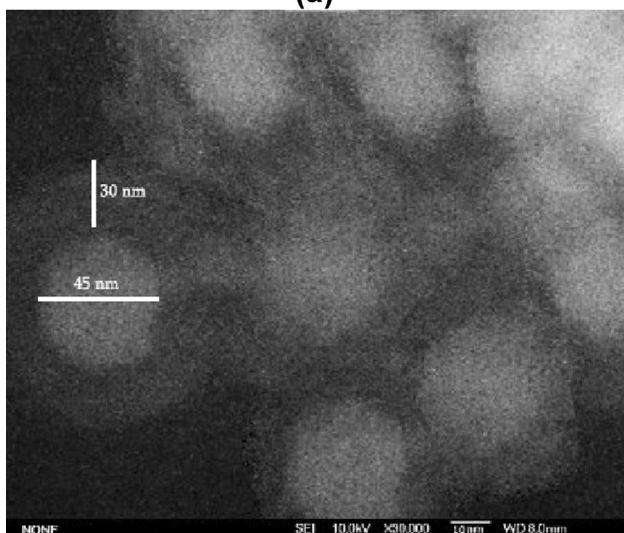
$$PV = \theta_{600} - \theta_{300} \quad (\text{cP}) \quad (2)$$

$$YP = 4.78(\theta_{300} - PV) \quad (\text{dyne/cm}^2) \quad (3)$$

In Eqs. (1–3),  $\theta_{300}$  and  $\theta_{600}$  are the pointer deviation of viscometer at the speeds of 300 and 600 rpm, respectively. Coefficient 4.78 in Eq. (3) is conversion factor from lb/100ft<sup>2</sup> to dyne/cm<sup>2</sup>.



(a)



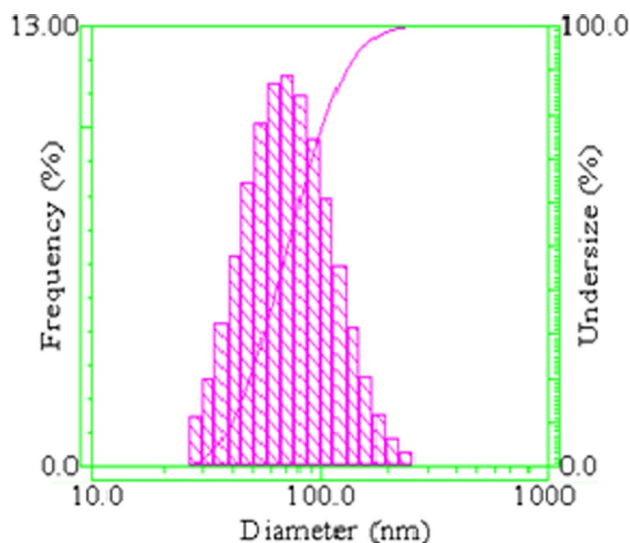
(b)

**Fig. 5** a SEM image of core-shell nanocomposite, b TEM image of core-shell nanocomposite

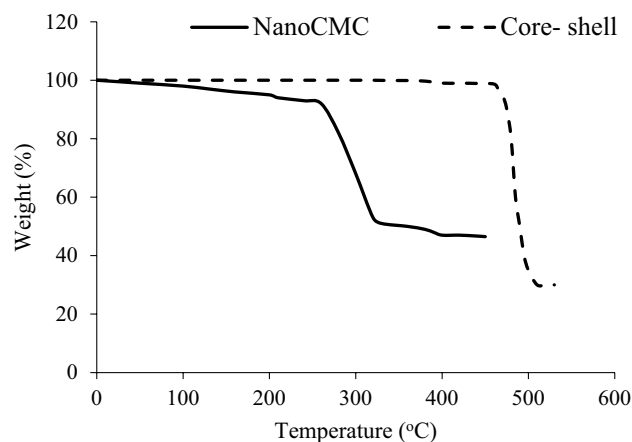
*Fluid loss and mud cake thickness* The filter press (100 psi pressure and room temperature) was used to measure the fluid loss and mud cake thickness of the drilling fluid samples after 30 min.

*Mud weight and pH* Mud balance and pH meter were used to measure weight and the acidic or basic properties of the drilling fluid samples, respectively.

All above tests (viscosity, fluid loss, cake thickness, mud weight and pH) were performed separately for three additives (core-shell nanocomposite, nanoCMC and bulk CMC). The results are presented and compared in the next section.



**Fig. 6** Particle size analyzer image of core-shell nanocomposite



**Fig. 7** Thermogravimetric curves of core-shell nanocomposite, nanoCMC and bulk CMC

## Results and discussion

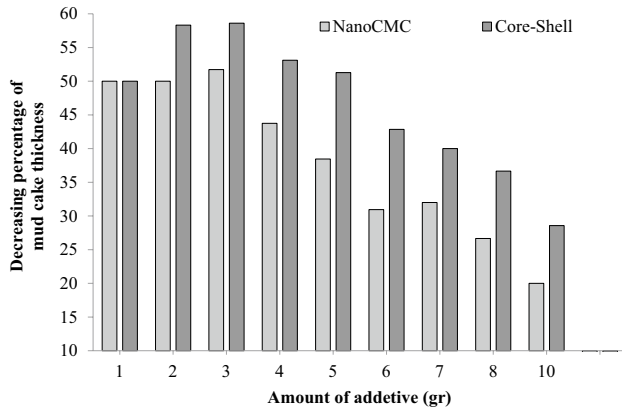
In this section, change in main drilling fluid properties as a result of addition of nanoCMC and core-shell nanocomposite is compared with bulk CMC as described in the previous section.

### Cake thickness

Table 1 compares cake thickness for addition of same amount of three additives. For a given amount of additive, core-shell nanocomposite gives minimum cake thickness. For a given additive, cake thickness increases with increasing amount of additive which is common due to static filtration conditions

**Table 1** Mud cake thickness of conventional CMC, CMC nanoparticle and core-shell nanocomposite

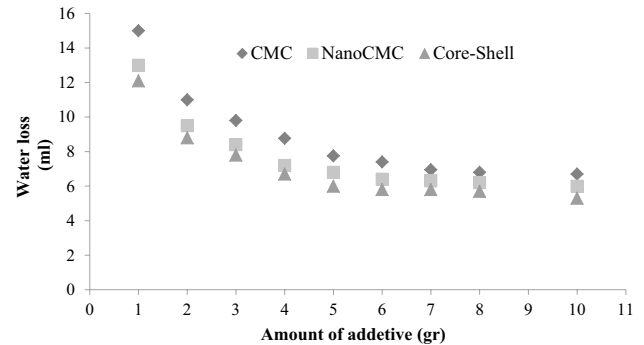
Type of additive	Amount of additive (gr)									
	1	2	3	4	5	6	7	8	10	
<i>Mud cake thickness (mm)</i>										
Convectional CMC	0.2	0.24	0.29	0.32	0.39	0.42	0.5	0.6	0.7	
CMC nanoparticle	0.1	0.12	0.14	0.18	0.24	0.29	0.34	0.44	0.56	
Core-shell nanocomposite	0.1	0.1	0.12	0.15	0.19	0.24	0.3	0.38	0.5	

**Fig. 8** Comparison of mud cake thickness resulted from addition of nanoCMC and core-shell nanocomposite (compared to bulk CMC)

(dead-end filtration in filter press and accumulation of suspended particles on the filter face); however, cake thickness grows more slowly for core-shell and nanoCMC compared to bulk CMC which shows improved performance. Figure 8 presents reduction in cake thickness (in percent) when different amounts of nanoCMC and core-shell nanocomposites were added into the drilling fluid in comparison with bulk CMC. Both nanoCMC and core-shell nanocomposite reduce cake thickness significantly compared to the bulk CMC. NanoCMC particles can reduce cake thickness 20–50% while almost 30–60% reduction can be achieved by adding core shell particles (i.e., thinner mud cake can be formed by using core shell nanoparticles, see Table 1). Minimum mud cake thickness can be obtained by adding 2–3 g (0.5 wt%) of both nanoparticles. Addition of 0.5 wt% nanocomposite can reduce almost 60% of cake thickness if compared with conventional CMC (about 50% for nanoCMC) which is a great achievement. Thinner cake is preferable during drilling of oil and gas wells due to common risk of pipe sticking problems.

## Fluid loss

Figure 9 compares fluid loss of drilling fluid prepared with bulk CMC, nanoCMC and core-shell nanocomposites additives. According to the results, fluid loss volume decreases significantly with increase in the amount of additive for bulk

**Fig. 9** Comparison of fluid loss of bulk CMC, nanoCMC and core-shell nanocomposite

CMC, nanoCMC and core-shell nanocomposite (almost same trend). The decrease is sharp up to almost 6 g additive and addition of more additive does not change fluid loss volume significantly. However, core-shell nanocomposite has the best performance with any given additive compared to nanoCMC and bulk CMC. For example, adding 4 g of three additives, core-shell nanocomposite decreases 22 and 31% more water loss than nanoCMC and bulk CMC, respectively.

The results show high performance of core-shell nanocomposites as drilling fluid additive for fluid loss control. According to Fig. 8, adding less than 5 g core-shell nanocomposite results in about 50% reduction in cake thickness and almost 27% prevention in fluid loss compared to bulk CMC.

Generally small particles in the external filter cake can cause lower cake permeability which also reduces fluid filtration (water loss) according to Darcy's law. It can be explained by filling of small pores in the mud cake with nanoparticles. On the other hand, in real drilling operations (dynamic filtration instead of static filtration in laboratory) where different force acting on a particle at the cake surface, lower filtration rate causes less normal drag force on the particle at the cake surface and thinner cake can be achieved due to tangential force exerted on a particle at the cake surface (Fig. 10, for detailed discussion and derivations see refs Kalantariasl et al. 2015a; Zinati et al.

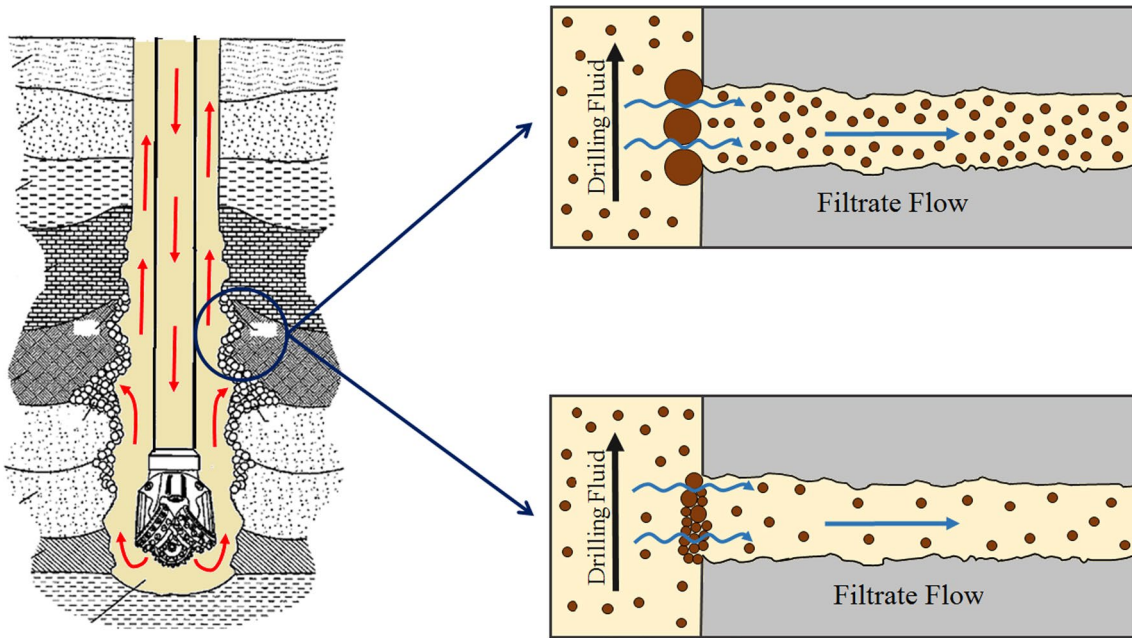


Fig. 10 Schematic: effect of particle size on fluid loss

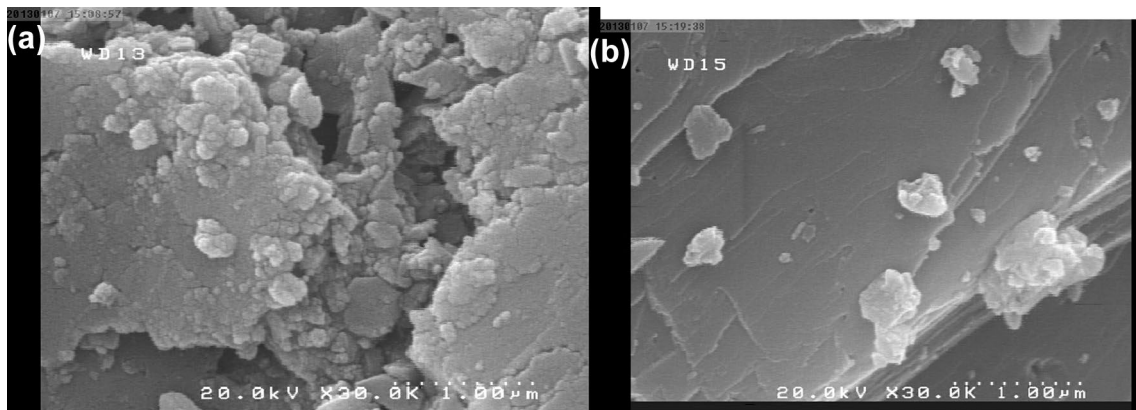


Fig. 11 SEM image of mud cake surface morphology after filtration test, a drilling fluid without core-shell nanocomposite, b drilling fluid +3gr core-shell nanocomposite

2009; Kalantariasl and Bedrikovetsky 2013; Kalantariasl et al. 2015b).

Figure 11 shows mud cake surface after filtration test with and without using nanocomposite (3 g) in drilling fluid. Based on this image, nanocomposite (Fig. 11b) in drilling fluid causes smoother cake surface compared to mud cake without nanocomposite, i.e., with conventional CMC (Fig. 11a).

**Rheological properties**

Plastic viscosity and apparent viscosity in the drilling fluid resulted from addition of bulk CMC, nanoCMC and

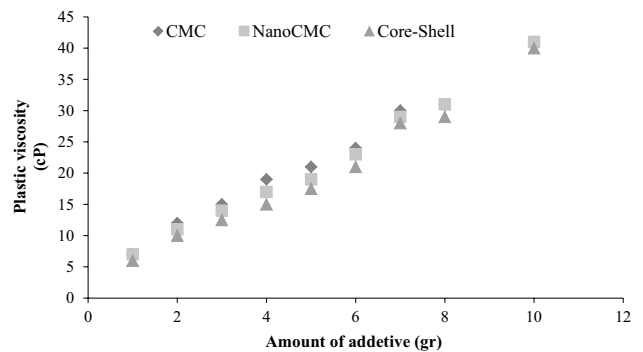


Fig. 12 Comparison of plastic viscosity of drilling fluid with bulk CMC, nanoCMC and core-shell nanocomposite additives

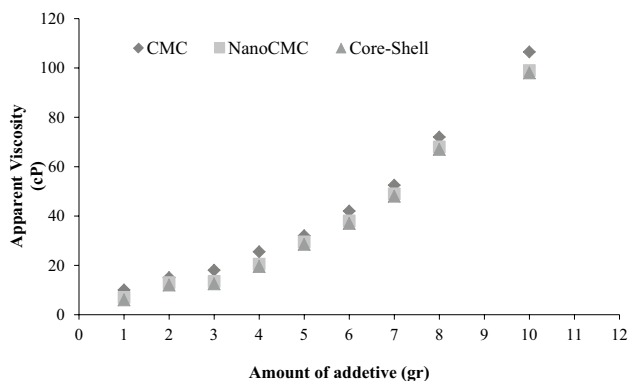


Fig. 13 Comparison of apparent viscosity of drilling fluid with bulk CMC, nanoCMC and core-shell nanocomposite additives

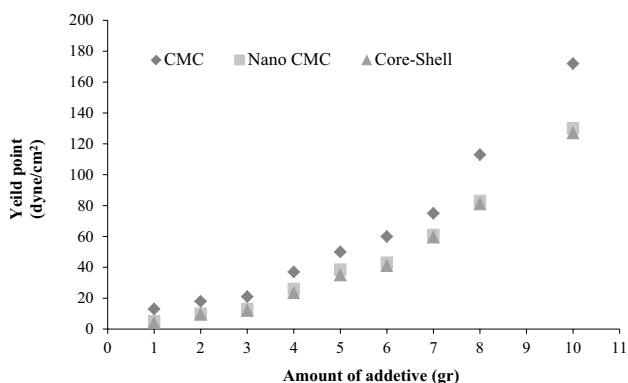


Fig. 14 Comparison of yield point of drilling fluid with bulk CMC, nanoCMC and core-shell nanocomposite additives

core-shell nanocomposite are present in Figs. 12 and 13, respectively. It shows that increase in the amount of bulk CMC, nanoCMC and core-shell nanocomposite increases both plastic and apparent viscosities for all three additives. Both plastic and apparent viscosities are almost insignificant to type of additives which is a positive sign for using core shell nanocomposite in drilling fluid. Conventional CMC (bulk CMC) is widely used in drilling oil and gas wells; however, core shell nanocomposite significantly reduces fluid loss and cake thickness with insignificant change in rheological properties compared to conventional CMC.

The results for yield point are shown in Fig. 14. It indicates that the yield point of bulk CMC is much greater than that of nanoCMC and nanocomposite. For example, yield points resulted from addition of 3 g bulk CMC, nanoCMC and nanocomposite are 21, 13 and 12 dyne/cm<sup>2</sup>, respectively. The yield point of core-shell nanocomposite is less than that of the nanoCMC (but very close) which can be due to difference in surface properties and interaction between particles as their size distribution is very close to each other (see Figs. 1, 6). Reduction in the yield point reduces the

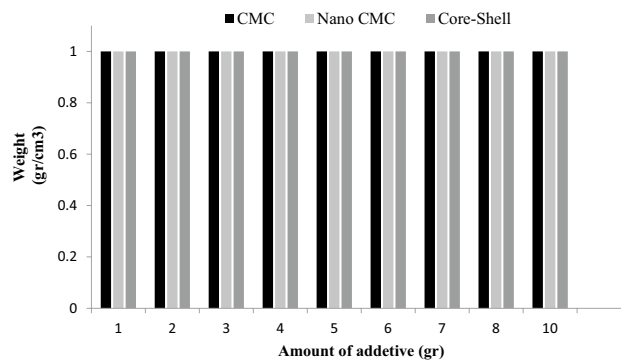


Fig. 15 Comparison of mud weight with bulk CMC, nanoCMC and core-shell nanocomposite additives

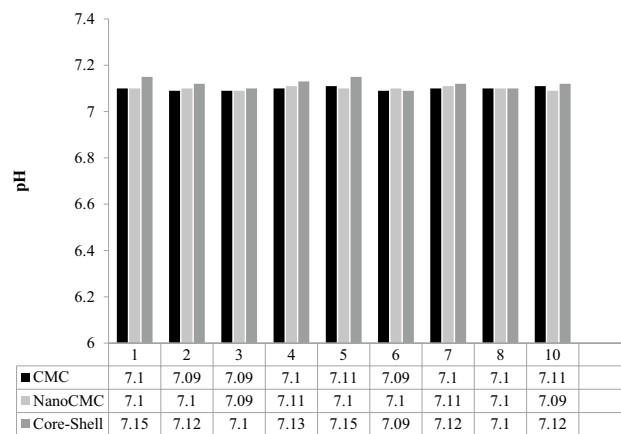


Fig. 16 Comparison of pH of drilling fluid with bulk CMC, nanoCMC and core-shell nanocomposite additives

power needed to pump the drilling fluid into the well and its circulation which is favorable in drilling fluid design.

### Mud weight and pH

Figures 15 and 16 show that adding bulk CMC, nanoCMC and core-shell nanocomposite to water-based drilling fluid has almost no effect on mud weight and pH which has another positive sign that addition of nanocomposite does not change weight and pH.

The results of this study show that addition of less than 4 g (< 1 wt%) core-shell nanocomposite significantly prevents fluid loss and creates thinner cake. Even, addition of 1 g (< 0.3 wt%) of core-shell nanocomposites to the water-based drilling fluid can reduce cake thickness by 50% if compared with conventional CMC (bulk CMC) additive (Fig. 8). Moreover, at the same time, it results in 24% less water loss into the formation (Fig. 9). The above values of cake thickness and fluid loss allow claiming of promising



results based on experimental data. Meanwhile, addition of low values of nanoparticles does not change viscosity and yield point significantly (see Figs. 11, 12, 13) if compared with conventional CMC which is used daily in drilling oil and gas wells. Reduction in yield point which is achieved by addition of nanocomposite can save energy for pumping and circulation of drilling fluid. The proposed additives can be used for fluid loss control when required in specific formations. The additives and their concentration can be used in dynamic and static core flooding which allows pressure drop monitoring and fluid loss volume measurement where obtained results can be used to recalculate cake permeability and thickness (Frequin et al. 2013; Keshavarz et al. 2015). High thermal stability of core–shell nanocomposites presented in this study makes it a favorable option for high-temperature formations which can be a subject for new investigation. Economic analysis of costs and benefits can be done for decision making on using nanoparticle additives for possible field applications. Effects of other parameters for real field applications can be also tested. For future research, instead of particle size analyzer (PSA), small-angle X-ray scattering (SAXS) can be used for measuring the nanoparticle size as it provides much more accurate information on size and size distribution (Borchert et al. 2005; Mozaffari et al. 2017). Moreover, effect of nanoparticles on drilling fluid rheology at high salinity and HPHT (high pressure high temperature) conditions can be investigated more deeply (Mozaffari et al. 2015; Sharma et al. 2016). Dynamic filtration of drilling fluid with nanoparticles highly improves our understanding of their effect when cross-flow filtration (instead of dead-end filtration) conditions can be simulated similar to drilling operations since stabilized cake thickness will be achieved and deposition and removal of nanoparticles significantly affect filtration properties (Kalantariasl and Bedrikovetsky 2013; Kalantariasl et al. 2014).

## Conclusions

In this work, core–shell nanocomposites were synthesized via miniemulsion polymerization method and characterized by SEM, TEM, PSA and TGA. The core–shell nanocomposite showed higher thermal stability than nanoCMC particles. The synthesized core–shell nanocomposite was added to the water-based drilling fluid, and its rheological and filtration properties were significantly improved compared with nanoCMC particles and conventional bulk CMC. The results indicate that:

Thinner cake can be obtained by adding core–shell nanocomposite into water-based drilling fluid even at small

amount. NanoCMC has the same trend but less effective than core–shell nanocomposite.

Core–shell nanocomposite caused significant reduction in fluid loss.

Yield point was decreased more by adding core–shell nanocomposite and nanoCMC if compared with bulk CMC.

Small change in fluid viscosity was observed as a result of adding core–shell nanocomposite to water-based drilling fluid.

Core–shell nanocomposite, nanoCMC and bulk CMC have no significant effect on weight and pH of water-based drilling fluid.

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