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# The influence of $\text{TiO}_2$ and $\text{SiO}_2$ nanoparticles on filtration properties of drilling muds

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Original scientific paper



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## Abstract

During the drilling of a well, differential pressure causes filtration of the mud liquid phase into the surrounding rocks. To reduce the filtrate invasion in the rock, it is necessary to maintain the density of the mud. Also, Wellbore Strengthening Materials (WSM) can be preventively added to the mud to increase the fracture initiation pressure of the drilled formation. The solid particles from the mud create a mud cake on the wellbore wall, which, for conventional drilling, implies the use of bentonite and barite whose particle dimensions range from 0.1 to 100  $\mu\text{m}$ . While drilling shales, rocks which contain pores that have an average pore size of 10 to 30 nm, it is not possible to create a high-quality mud cake, so water passes into the shale and creates wellbore instability problems. To stabilize the shale, the petroleum industry uses oil-based muds, but due to their environmental impact, it is not always possible to implement them. Nanoparticles, because of their small dimensions, can enter into the nanopores and fill this space and strengthen the rock, resulting in decreased filtration. In this paper, a review of previous laboratory research of adding nanoparticles on filtration is shown. Also, the impact of adding nanoparticles on API and HTHP filtration is examined by adding  $\text{TiO}_2$  and  $\text{SiO}_2$  nanoparticles in concentrations of 0.5 wt% and 1 wt% to the five water-based muds. The best result was obtained with  $\text{TiO}_2$  nanoparticles at a concentration of 0.5 wt%.

## Keywords

$\text{TiO}_2$  and  $\text{SiO}_2$  nanoparticles, mud, filtration, wellbore stability

## 1. Introduction

Nanofluids are defined as suspensions of nanoparticles having an average particle diameter up to 100 nm (Ilyas et al., 2014). Material properties at a nanometer scale can be significantly different from a material which has larger dimensions. The nanoparticles have a much more specific surface (see Figure 1) compared to the specific surface of a material which has larger dimensions, for the same unit of mass, resulting in greater chemical reactivity and a greater influence on their physical properties (Aftab et al., 2017; Kök and Bal, 2019). Due to the larger specific surface, nanoparticles can be linked in a way to create firmer and lighter materials (El-Diasty and Ragab, 2013).

During drilling, companies face many problems that must be resolved in order to enable safe drilling progress in accordance with the project. The basic function of drilling mud is to carry out cuttings from a wellbore while maintaining the desired bottom hole pressure (BHP) and wellbore stability (Al-Zubaidi et al., 2017; Zadavec and Krištafor, 2018). Bottomhole pressure at a certain depth is primarily dependent on the density of

the drilling mud and during conventional drilling, it must be higher than the expected pore pressure. During the drilling of a well, differential pressure, which is the difference between the pressure in the well and the pore pressure at a certain depth, causes the filtration of the mud liquid phase into the surrounding rocks. At the same time, the solid particles from the mud create a mud cake on the wellbore wall. By increasing the pressure in the borehole above the fracturing pressure of the rock results in the occurrence of fractures and fissures. (Pašić, 2012). In order to reduce the filtrate invasion in the rock during the drilling of wells, certain preventive measures should be taken. First, it is necessary to prepare a mud which has a certain density that will not cause a pressure

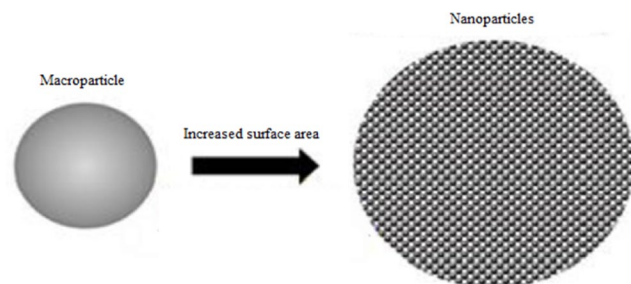


Figure 1: Increasing the specific surface by dividing macroparticles to nanoparticles (Salih et al., 2016)

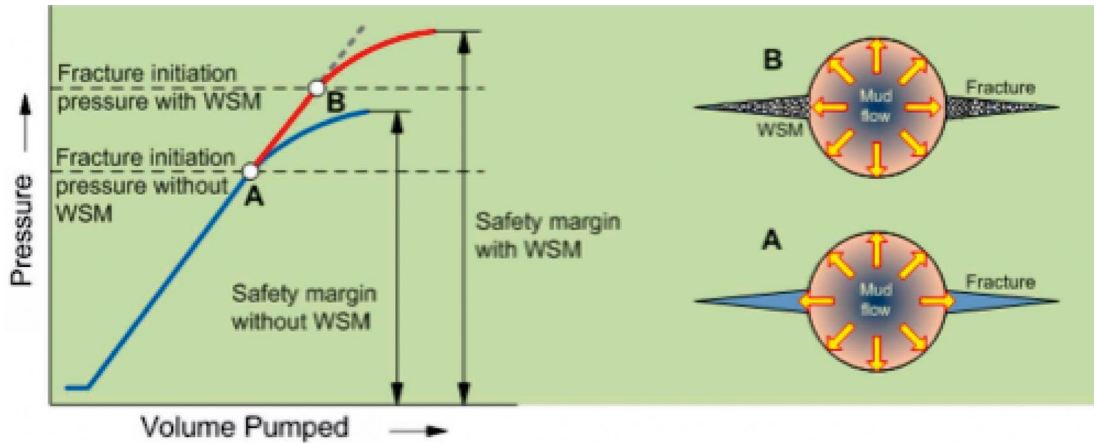


Figure 2: Wellbore strengthening materials increase the fracture initiation pressure (Gaurina-Međimurec et al., 2015)

Table 1: Types of rocks, permeability, pore size and materials used for plugging pores (Amanullah and Al-Tahini, 2009)

Type of rock	Permeability, $\mu\text{m}^2$	Pore size, $\mu\text{m}$	Material used for plugging pores
Shale	$10^{-9}$	< 1	Nanoparticles
Silt	$10^{-6}$	1 – 10	Nano and microparticles
Consolidated sand	$10^{-3}$	10 – 100	Nano and microparticles
Unconsolidated sand	1	> 100	Nano, micro and macroparticles

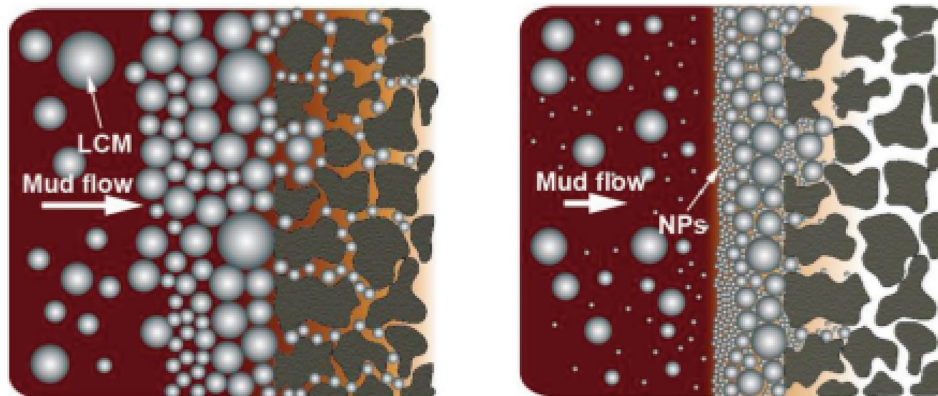


Figure 3: The action of nanoparticles to reduce filtration and increase the stability of the wellbore (Contreras et al., 2014b)

in the wellbore higher than the fracture pressure of the formation. Additionally, Wellbore Strengthening Materials (WSM) are preventively added to the mud in order to close the existing natural cracks and thereby increase the rock strength or fracture pressure of these hardened rocks (see Figure 2). WSM are very similar to Lost Circulation Materials (LCM).

According to Figure 2, case A shows a fracture initiation pressure without added WSM, while case B shows a fracture initiation pressure with WSM. In the case when WSM are not used (A), fracture initiation pressure is lower, and for further drilling, a lower density mud is allowed than in the case where WSM are added to the drilling mud (B). Wellbore strengthening can be applied

in cases where the borehole is made in areas a) where frequent mud losses occur, b) when there is a small difference between the hydrostatic pressure in the well and the fracture pressure, c) for drilling wells in depleted reservoirs, etc. (Gaurina-Međimurec et al., 2015). In recent years, the drilling of unconventional reservoirs is rapidly rising, preferably shales containing nanopores having an average size of 10 to 30 nm and they are characterized by their extremely low permeability (Sensoy et al., 2009). Due to this, it is not possible to create a high-quality mud cake on such rocks by using conventional mud additives. Conventional mud additives, such as bentonite and barite, have particle diameters ranging from 0.1 to 100  $\mu\text{m}$ . Consequently, water passes into the

**Table 2:** The type, size and concentration of nanoparticles used in different laboratory research

Reference	Type of nanoparticle	Size (nm)	Concentration of nanoparticles (wt%)	Mud type and formulation	Type of filtration test and conditions	Impact of adding nanoparticles on filtration	The highest measured reduction /increase of filtration (%)
Sarivatsa and Ziaja, 2011	$SiO_2$	N/A	<ul style="list-style-type: none"> <li>• 10</li> <li>• 20</li> <li>• 30</li> </ul>	<ul style="list-style-type: none"> <li>• Surfactant-polymer based mud</li> </ul>	<ul style="list-style-type: none"> <li>• API</li> <li>• PPT (<math>\Delta p=69</math> bar, <math>T=93</math> °C), ceramic disc 775 mD</li> </ul>	reduces filtration	40
Li et al., 2012	new nano additive (unknown composition)	N/A	<ul style="list-style-type: none"> <li>• 3</li> </ul>	<ul style="list-style-type: none"> <li>• Base mud (unknown composition)</li> </ul>	<ul style="list-style-type: none"> <li>• API filtration</li> </ul>	reduces filtration	25
Contreras et al., 2014a	in-house prepared iron-based and calcium-based nanoparticles	N/A	<ul style="list-style-type: none"> <li>• 0.5</li> <li>• 1</li> <li>• 2.5</li> </ul>	<ul style="list-style-type: none"> <li>• Oil-based mud (Oil/Water Ratio 90:10) emulsifier, <math>CaCl_2</math> brine, hydrated lime, gilsonit, organophilic clay)</li> </ul>	<ul style="list-style-type: none"> <li>• API filtration</li> <li>• PPT filtration (<math>\Delta p=34.5</math> bar, <math>T=121</math> °C), ceramic disc 775 mD</li> </ul>	reduces filtration	90
Fakoya and Shah, 2014	$SiO_2$	20	<ul style="list-style-type: none"> <li>• 0.058</li> <li>• 0.24</li> <li>• 0.4</li> </ul>	<ul style="list-style-type: none"> <li>• Surfactant-based, Polymer-based and Surfactant-polymer based mud (4% KCl solution, surfactant, guar, polyanionic cellulose (PAC))</li> </ul>	<ul style="list-style-type: none"> <li>• API filtration</li> </ul>	reduces filtration	93.9
Vryzas et al., 2015	powder $Fe_2O_3$ and powder $SiO_2$	$Fe_2O_3 <50$ ; $SiO_2$ 12	<ul style="list-style-type: none"> <li>• 0.5</li> <li>• 1.5</li> <li>• 2.5</li> </ul>	<ul style="list-style-type: none"> <li>• Water-based mud (7 % (w/w) bentonite suspension)</li> </ul>	<ul style="list-style-type: none"> <li>• API filtration</li> <li>• HTHP filtration (<math>\Delta p=20.7</math> bar, <math>T=121</math> °C)</li> </ul>	$Fe_2O_3$ reduces filtration, $SiO_2$ increases filtration	42.5 reduction; 57.8 increase
Taragikhah et al., 2015	$SiO_2$	N/A	<ul style="list-style-type: none"> <li>• 0.5</li> <li>• 1</li> <li>• 2</li> </ul>	<ul style="list-style-type: none"> <li>• Water-based mud (salt, viscosifier, fluid loss controller)</li> </ul>	<ul style="list-style-type: none"> <li>• API filtration</li> </ul>	Silica does not affect API filtration below 1 wt%, at higher concentrations filtration increase	115 increase at higher concentrations
Wahid et al., 2015	$SiO_2$ powder	10-20	<ul style="list-style-type: none"> <li>• 0.16-1.05</li> </ul>	<ul style="list-style-type: none"> <li>• Synthetic-based mud (base oil, emulsifier, viscosifier, fluid loss control, lime, <math>CaCl_2</math>, barite, drill solids)</li> </ul>	<ul style="list-style-type: none"> <li>• HTHP filtration (<math>T=135</math> °C and <math>176.7</math> °C)</li> </ul>	reduces filtration	41.67
Krishnan et al., 2016	boron-based nanomaterial enhanced additive	N/A	<ul style="list-style-type: none"> <li>• 1-5 vol%</li> </ul>	<ul style="list-style-type: none"> <li>• Water-based mud (density <math>1200</math> <math>kg/m^3</math> and <math>1620</math> <math>kg/m^3</math>)</li> </ul>	<ul style="list-style-type: none"> <li>• HTHP filtration (<math>\Delta p=34.5</math> bar, <math>T=150</math> °C)</li> </ul>	in $1200$ $kg/m^3$ reduces filtration, in $1620$ $kg/m^3$ no effect	50

shale pores whose ions react with the clay minerals resulting in wellbore instability problems (Pašić et al., 2017). To stabilize the borehole wall during the drilling of shale formations, the petroleum industry uses oil-based muds that do not react with clay minerals in the shale, but due to their environmental impact, it is not always possible to implement them (Sensoy et al.,

2009). Therefore, the use of nanoparticles is considered, which, due to their small dimensions, can enter into the nanopores of shale, fill this space and strengthen the rock, which also results in an increased fracture initiation pressure. Nanofluids are defined as suspensions of nanoparticles which have an average particle diameter less than 100 nm (Ilyas et al., 2014). Table 1 shows dif-

Table 2: Continued

Reference	Type of nanoparticle	Size (nm)	Concentration of nanoparticles (wt%)	Mud type and formulation	Type of filtration test and conditions	Impact of adding nanoparticles on filtration	The highest measured reduction/increase of filtration (%)
Mahmoud et al., 2016	Fe <sub>2</sub> O <sub>3</sub> and SiO <sub>2</sub> powder	Fe <sub>2</sub> O <sub>3</sub> <50 and SiO <sub>2</sub> 12	<ul style="list-style-type: none"> <li>• 0.3</li> <li>• 0.5</li> <li>• 1.5</li> <li>• 2.5</li> </ul>	<ul style="list-style-type: none"> <li>• Water-based mud</li> <li>• (7 % (w/w) bentonite suspension)</li> </ul>	<ul style="list-style-type: none"> <li>• HTHP filtration (<math>\Delta p=13.8-34.5</math> bar, <math>T=79.5-176.7</math> °C)</li> <li>• dynamic filtration</li> </ul>	Fe <sub>2</sub> O <sub>3</sub> reduces filtration, SiO <sub>2</sub> increases filtration	42.5 reduction; 57.8% increase; dynamic increase by 79.71
Salih et al., 2016	SiO <sub>2</sub>	N/A	<ul style="list-style-type: none"> <li>• 0.1</li> <li>• 0.3</li> <li>• 0.5</li> <li>• 0.7</li> </ul>	<ul style="list-style-type: none"> <li>• Water-based mud (bentonite 5.6 wt%, barite 5.2 wt%, NaOH)</li> </ul>	<ul style="list-style-type: none"> <li>• API filtration</li> </ul>	reduces filtration	44
Salih and Bilgesu, 2017	nanosilica, nanotitanium, nanoaluminum	N/A	<ul style="list-style-type: none"> <li>• 0.1</li> <li>• 0.3</li> <li>• 0.5</li> <li>• 0.7</li> </ul>	<ul style="list-style-type: none"> <li>• Water-based mud (bentonite 5.6 wt%, barite 5.2 wt%, 0.5 g NaOH)</li> </ul>	<ul style="list-style-type: none"> <li>• API filtration</li> </ul>	Nanosilica and nanotitanium reduce filtration while nanoaluminum does not affect API filtration	44 nanosilica; 20 nanotitanium
Loggins et al., 2017	barite nanoparticles (BaSO <sub>4</sub> )	50	<ul style="list-style-type: none"> <li>• 1.5</li> <li>• 3</li> </ul>	<ul style="list-style-type: none"> <li>• Water-based mud (water 330 ml, 1-hexadecene 20 ml, SDS, starch, PAC-R, KCl, NaCl, barite)</li> </ul>	<ul style="list-style-type: none"> <li>• API filtration</li> </ul>	reduces filtration	54
Vryzas et al., 2017	Fe <sub>3</sub> O <sub>4</sub>	N/A	<ul style="list-style-type: none"> <li>• 0.5</li> </ul>	<ul style="list-style-type: none"> <li>• Water-based mud (Na-bentonite 7 wt%)</li> </ul>	<ul style="list-style-type: none"> <li>• HTHP filtration (<math>\Delta p=34.5</math> bar, <math>T=121</math> °C)</li> </ul>	reduces filtration	47
Alvi et al., 2018	Boron Nitride and Fe <sub>2</sub> O <sub>3</sub>	250	<ul style="list-style-type: none"> <li>• 0.0095, 0.019, 0.38 (Fe<sub>2</sub>O<sub>3</sub>)</li> <li>• 0.0095, 0.019, 0.0284, 0.038 (BN)</li> </ul>	<ul style="list-style-type: none"> <li>• Water-based mud (500 ml water, 0.5 g xanthan biopolymer, 2.5 g KCl, 25 g bentonite)</li> </ul>	<ul style="list-style-type: none"> <li>• API filtration</li> </ul>	Boron Nitride and Fe <sub>2</sub> O <sub>3</sub> no significant effect	Boron Nitride increase 7.1, Fe <sub>2</sub> O <sub>3</sub> decrease 14.3 for concentration 0.0095 wt%, but at higher concentrations decrease by 10.7
Mahmoud et al., 2018	Fe <sub>2</sub> O <sub>3</sub>	<50	<ul style="list-style-type: none"> <li>• 0.3</li> <li>• 0.5</li> <li>• 1</li> </ul>	<ul style="list-style-type: none"> <li>• Water-based mud (Ca bentonite 7 wt%, hyperbranched polymer, polyanionic cellulose, lignosulfonate-based thinner, NaOH, CaCO<sub>3</sub>, Mn<sub>3</sub>O<sub>4</sub>)</li> </ul>	<ul style="list-style-type: none"> <li>• HTHP filtration (<math>\Delta p=20.7</math> bar, <math>T=121</math> °C)</li> </ul>	Up to concentration of 0.5 wt% reduces filtration, concentration of 1 wt% increases filtration	16,9 decrease at concentration of 0.3 wt%, 15.6 increase at concentration of 1 wt%

ferent types of rocks, common permeability, pore dimensions, and materials used for the effective plugging of the pores, while the effect of adding nanoparticles to reduce filtration and increase the stability of the wellbore is shown in **Figure 3**.

Due to the pressure difference between hydrostatic/hydrodynamic and pore pressure at a certain depth, there

is still a penetration flow of mud from the wellbore into the rock. This problem is present during drilling unconventional as well as conventional reservoirs. **Figure 3 (left)** shows the case when conventional lost circulation materials were added to mud to fill the pores on the borehole walls.



**Table 3:** Data on TiO<sub>2</sub> and SiO<sub>2</sub> nanoparticles (Evonik Industries, 2017)

Brand name	Aerodisp W740X	Aerodisp W7330N
Nanoparticles type	TiO <sub>2</sub>	SiO <sub>2</sub>
Nanoparticles content (wt%)	39-41	30
Appearance	an aqueous suspension of TiO <sub>2</sub> nanoparticles	an aqueous suspension of SiO <sub>2</sub> nanoparticles
Density @20°C (kg/m <sup>3</sup> )	1410	1200
Stabilizing agent	-	NaOH
Average particle size (D50)	70 nm	120 nm
pH	5-7	9.5-10.5

As seen in **Figure 3 (left)**, LCM has larger particle dimensions than pores in formations and cannot adequately plug them. By adding nanoparticles, they plug the space between larger particles which results in the creation of a high quality, thin, impermeable mud cake (**Figure 3 (right)**).

According to literature, the advantage of adding nanoparticles to reduce filtration was determined by laboratory tests with only a few field applications. For example, during the drilling of a 12 1/4" interval through a freshwater zone in the Reconcavo Basin, Brazil, there was a risk of contamination of the freshwater zone. To prevent contamination of the freshwater zone, Nano Fluid without chlorides was used. This type of mud also provides adequate shale inhibition as well as optimum drilling performance. After a 13 3/8" casing was run to a TD of 515 m, drilling was resumed with the Nano Fluid from 515 m to 1600 m without any problems, so the Nano Fluid showed excellent performance while drilling the shale formation (**Barroso et al., 2018**). A detailed summary of the most relevant laboratory research related to nanoparticle application for reducing the filtration of drilling fluids is presented in the next section.

## 2. Review of previous laboratory tests

A summary review of the type, size and concentration of nanoparticles used to reduce filtration and improve wellbore strength is shown in **Table 2**. Most of presented papers indicate the possibility of nanoparticles to plug the nanopores, which reduces filtration and improves wellbore strength.

By analyzing the results of previous tests, the addition of nanoparticles to some muds gave unexpected results. For example, **Vryzas et al. (2015)** found that iron oxide nanoparticles significantly reduced the filtration of bentonite suspensions. In high temperature high pressure (HTHP) conditions, the largest filtration reduction (reduction was 42.5% in comparison to bentonite suspension without nanoparticles) was observed when iron nanoparticles at a concentration of 0.5 wt.% were added into bentonite suspensions. Also, API filtration was measured and at higher concentrations (1.5 wt% and 2.5 wt%), the results showed a decrease in filtration. At the same time, both API filtration and HTHP filtration were

significantly increased with the addition of SiO<sub>2</sub> nanoparticles at all three concentrations (0.5 wt%, 1.5 wt% and 2.5 wt%). **Taraghikhah et al. (2015)** found that filtration properties remained unaltered at concentrations below 1 wt% of SiO<sub>2</sub> nanoparticles in comparison to mud without nanoparticles. **Wahid et al. (2015)** added SiO<sub>2</sub> nanoparticles in a synthetic mud at a concentration of 0.32 to 0.71 wt% and obtained a reduction in the filtration and thickness of the mud cake, with stable rheological properties.

According to the results of laboratory research presented in **Table 2**, it is very hard to extract specific conclusions. Despite the presented test types and conditions, in most cases there is a lack of detailed composition of the tested fluids (concentration of added additives, nanoparticle size, nanoparticle and additives manufacturer, etc.) which greatly aggravates a quality comparison of the presented results. All of this indicates that there is still room for future examinations and that the impact of nanoparticle addition into mud is still partially unclear.

## 3. Material and methods

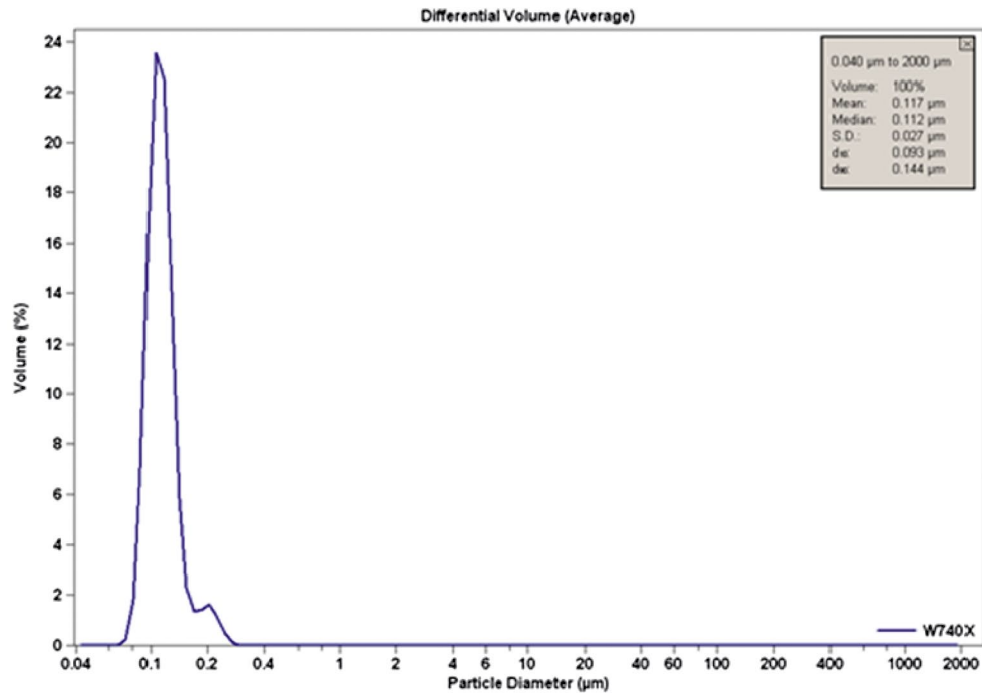
The impact of adding nanoparticles to the water-based mud on its filtration properties was examined at the Drilling Fluid Laboratory (Department for Petroleum Engineering, Faculty of Mining, Geology and Petroleum Engineering, University of Zagreb) in Zagreb, Croatia. Data on TiO<sub>2</sub> and SiO<sub>2</sub> nanoparticles obtained from the manufacturers are shown in **Table 3 (Evonik Industries, 2017)**.

In order to gain a better understanding of the effect of adding nanoparticles on the filtration properties of drilling mud, five water-based drilling mud samples were prepared and tested: base bentonite drilling mud (A) and four samples containing different types of nanoparticles (B-E) (see **Table 4**).

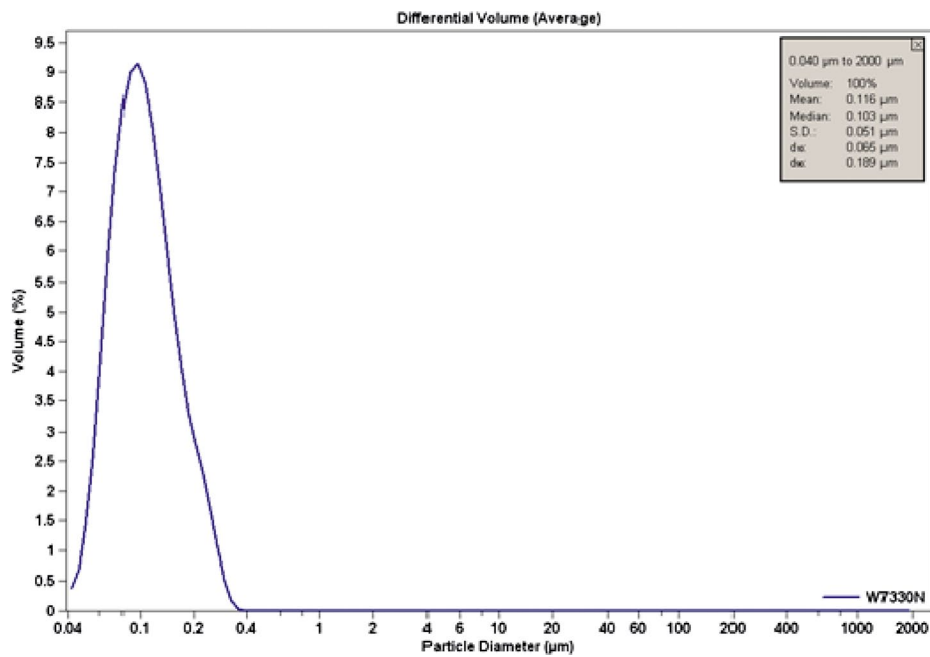
After the preparation of the bentonite drilling mud (A), 9 or 18 ml of Aerodisp W740X (TiO<sub>2</sub>) nanoparticles were added to a base mud which approximately corresponds to the concentration of 0.5 wt% and 1 wt% of TiO<sub>2</sub> nanoparticles (mud B and C). Muds were stirred for 30 minutes in a stirrer to reduce the possibility of agglomeration. After the drilling mud sample was prepared, the influence of adding TiO<sub>2</sub> on the filtration prop-

**Table 4:** Composition of tested drilling muds

Composition	Unit	Drilling muds				
		A	B	C	D	E
Water	ml	1000	1000	1000	1000	1000
Bentonite	g	30	30	30	30	30
NaOH	g	2	2	2	2	2
PAC LV	g	2	2	2	2	2
Aerodisp W740X	ml	-	9	18	-	-
Aerodisp W7330N	ml	-	-	-	14	28



**Figure 4:** Granulometric curve of Aerodisp W740X



**Figure 5:** Granulometric curve of Aerodisp W7330N

**Table 5:** Influence of adding TiO<sub>2</sub> and SiO<sub>2</sub> and nanoparticles on API filtration (according to Mijić et al., 2017)

Time, min	Drilling muds				
	A	B	C	D	E
	Concentration of nanoparticles				
	0	0.5 wt% TiO <sub>2</sub>	1.0 wt% TiO <sub>2</sub>	0.5 wt% SiO <sub>2</sub>	1.0 wt% SiO <sub>2</sub>
	Fluid Loss (ml)				
1	2	2.5	2.5	2	2
5	5	5.25	5.5	4.75	4.75
7.5	6	6.5	6.5	5.75	6
10	7	7.5	7.5	6.75	7
15	8.5	8.75	9.5	8.5	8.5
20	10	10.5	11	10	10
25	11	11.5	12	11	11.5
30	12	12.5	13.25	12.5	12.5

erties and mud cake thickness were determined. In the same manner by adding 14 ml (0.5 wt%) or 28 ml (1 wt%) Aerodisp W7330N (SiO<sub>2</sub>) nanoparticles into bentonite drilling mud (A), muds D and E were prepared.

### 3.1. Characterization of nanoparticles

To verify the data obtained from the manufacturer, granulometric analyses of the samples were made at Ruđer Bošković Institute, Zagreb, Croatia to determine particle size distribution. Two samples were analyzed, namely:

1. Aerodisp W740X - an aqueous suspension of TiO<sub>2</sub> nanoparticles and
2. Aerodisp W7330N - an aqueous suspension of SiO<sub>2</sub> nanoparticles.

The selected samples were diluted with deionized water prior to the granulometric analysis to achieve the optimum suspension concentration. Granulometric analyses were done by the laser beaming method using the instrument LS 13320 (Beckman Coulter). A ULM module (universal fluid module) was used for the analysis, ranging from 0.04 to 2000 µm. Each sample was measured in triplicate. **Figures 4** and **5** show the average result of two consecutive measurements.

According to the shape of the granulometric curve, it can be concluded that Aerodisp W740X – an aqueous suspension of TiO<sub>2</sub> nanoparticle has a narrow and uniform distribution and these particles are less than 300 nm, while the average particle size is 117 nm. Aerodisp W7330N – an aqueous suspension of SiO<sub>2</sub> nanoparticles has a wider but relatively symmetric distribution and these particles are less than 400 nm, while the average particle size is 116 nm. Although the manufacturer declared an average particle size of 70 nm for TiO<sub>2</sub> nanoparticles and 120 nm for SiO<sub>2</sub> nanoparticles, these measurements showed that both types of the implemented nanoparticles had similar average particle size. This

means that these two types of nanoparticles can be comparable in this research.

## 4. Results of filtration of tested drilling muds

### API filtration

The API filtration properties of selected drilling muds were determined according to the **API RP 13B-1 (1997)** standard. The API filter press consists of a cell to which a pressure of 6.895 bar (100 psi) was applied for a period of 30 minutes to measure the volume of filtrate extracted from the drilling mud through a filter paper (Whatman No. 50) and gathered in a measuring cylinder. The filter paper surface, and thus the filtration area is 45.8 cm<sup>2</sup> (7.1 in<sup>2</sup>).

Based on the results of the API filtration, it can be seen that, regardless of the type and concentration of nanoparticles TiO<sub>2</sub> and SiO<sub>2</sub>, their addition to the base drilling mud caused an increase in the API filtration value from 4.17% (muds B, D and E) to 10.42% (mud C) (see **Table 5**).






Bentonite drilling mud with added TiO<sub>2</sub> nanoparticles at a concentration of 0.5 wt% (mud B) has an increased API filtration value by 4.17 % in comparison to the value measured with bentonite drilling mud (mud A) and the same results were obtained with mud D and mud E. A more significant difference was made with the addition of TiO<sub>2</sub> nanoparticles to the bentonite drilling mud at a concentration of 1 wt% (mud C). It increased the API filtration value by 10.42% in comparison to the bentonite drilling mud (mud A).

### HTHP filtration






The HTHP filter press consists of a cell in which a high pressure and high temperature are applied, and the filter medium can be filter paper (Whatman, No. 50) or a



**Table 6:** Muds filtration results from PPT for a ceramic disc with permeability of  $0.4 \mu\text{m}^2$  (400 mD)

	Disc permeability – $0.4 \mu\text{m}^2$ (400 mD)				
	Differential pressure - 55 bar (800 psi)				
	Temperature – 88 °C (192 °F)				
Mud	A	B	C	D	E
Concentration of nanoparticles	0	0.5 wt% $\text{TiO}_2$	1.0 wt% $\text{TiO}_2$	0.5 wt% $\text{SiO}_2$	1.0 wt% $\text{SiO}_2$
$V_{7.5}$	9.5	9.5	13	11	11
$V_{30}$	18	14	19	17	19
PPT filtrate volume, ml	36	28	38	34	38
Spurt loss, ml	2	10	14	10	6
Ceramic disc with mud cake					

**Table 7:** Muds filtration results from PPT for a ceramic disc with permeability of  $0.75 \mu\text{m}^2$  (750 mD)

	Disc permeability - $0.75 \mu\text{m}^2$ (750 mD)				
	Differential pressure - 55 bar (800 psi)				
	Temperature – 88°C (192°F)				
Mud	A	B	C	D	E
Concentration of nanoparticles	0	0.5 wt% $\text{TiO}_2$	1.0 wt% $\text{TiO}_2$	0.5 wt% $\text{SiO}_2$	1.0 wt% $\text{SiO}_2$
$V_{7.5}$	15.5	13	15	13	17
$V_{30}$	23.5	19.5	23.5	21	25
PPT filtrate volume, ml	47	39	47	42	50
Spurt loss, ml	15	13	13	16	18
Ceramic disc with mud cake					

ceramic disk. The filtration area is  $22.9 \text{ cm}^2$  ( $3.5 \text{ in}^2$ ), twice as low as that of API filtration. In this paper, the test was carried out on a device for determining the ability of the drilling fluid to plug pores in a ceramic disc called a Permeability Plugging Tester (PPT), which represents a modification of the standard HTHP filter press. This device is used for conducting filtration tests under high pressure and high temperature conditions and is useful for assessing the ability of the drilling mud to form a high-quality mud cake due to the size and concentration of the solid particles present in drilling mud, which will prevent the further penetration of the filtrate through the filter medium. The test was carried out with the base drilling mud (mud A) and the drilling muds which contain  $\text{TiO}_2$  (muds B and C) and  $\text{SiO}_2$  (muds D and E) nanoparticles added in concentrations of 0.5 and 1 wt%. The permeabilities of the used ceramic discs are  $0.4 \mu\text{m}^2$  (400 mD) and  $0.75 \mu\text{m}^2$  (750 mD) and the tests were carried out at a differential pressure of 55 bar (800 psi) and a temperature of  $88^\circ\text{C}$  ( $192^\circ\text{F}$ ). Ceramic disc

permeabilities for this study are selected based on the summary of the laboratory research presented in **Table 2**. Test results obtained using the PPT device are shown in **Tables 6** and **7**. Since the filtration surface in the API filtration is twice as large, to compare the results of the PPT test to an API filtration test, fluid volume collected after 30 minutes needs to be multiplied by 2 (see **Equation 1**), while the initial filtration or spurt loss can be calculated using **Equation 2 (OFI Testing Equipment, Inc., 2014)**:

$$\text{PPT filtrate volume} = 2 \cdot V_{30} \quad (1)$$

$$\text{Spurt loss} = 4 \cdot V_{7.5} - 2 \cdot V_{30} \quad (2)$$

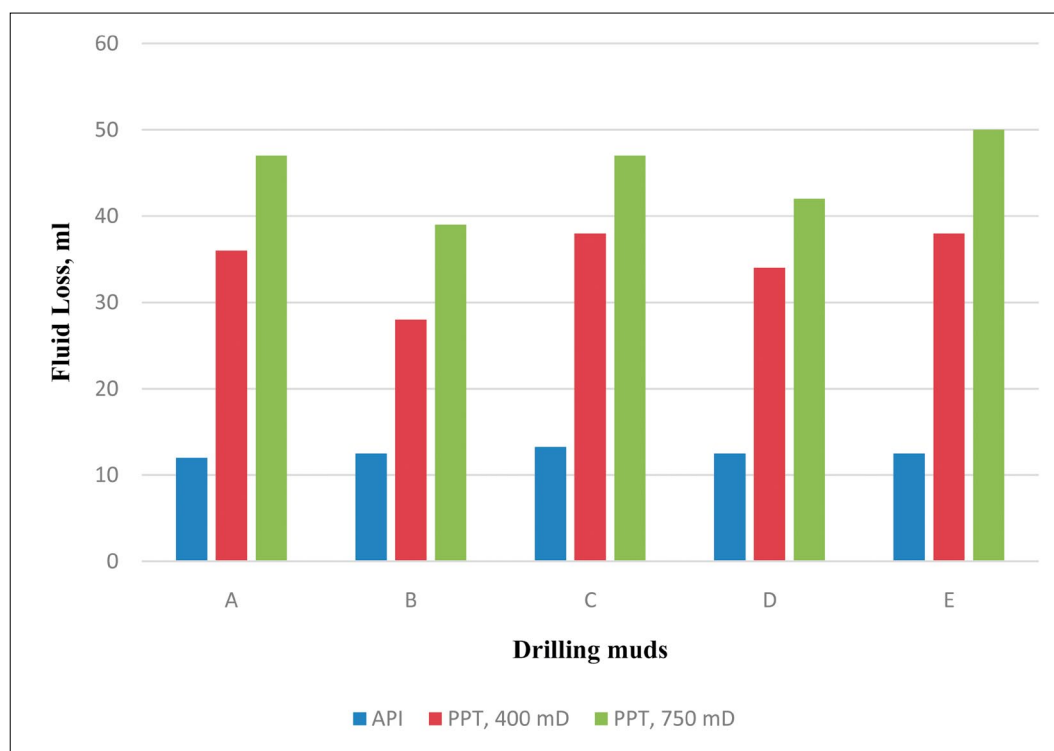
where:

PPT filtrate volume, ml

$V_{7.5}$  – fluid volume collected after 7.5 minutes, ml

$V_{30}$  – fluid volume collected after 30 minutes, ml

Spurt loss – fluid volume collected before forming a mud cake, ml



**Figure 6:** Comparison of filtration for all tested drilling muds

Filtration through the  $0.4 \mu\text{m}^2$  (400 mD) ceramic disc showed a slight increase in filtration volume (after 30 minutes it was only 5.56% compared to the base drilling mud) for mud containing  $\text{TiO}_2$  nanoparticles at a concentration of 1 wt% (mud C) and mud containing  $\text{SiO}_2$  nanoparticles at a concentration of 1 wt% (mud E). Those results correspond to results observed with the API filtration measurements for muds C and E. Filtration for mud containing  $\text{TiO}_2$  nanoparticles at a concentration of 0.5 wt% (mud B) was decreased by 22.2% in comparison to the base mud, while the addition of  $\text{SiO}_2$  nanoparticles at a concentration of 0.5 wt% to the base mud (mud D) resulted in a filtration volume reduction after 30 minutes of only 5.6%. However, observing the spurt loss or the amount of fluid that penetrates through the disc before the mud cake is formed, it can be seen that the amount of fluid that is lost before the formation of the mud cake is significantly higher for all the tested muds than in the case of the base drilling mud (mud A). Keeping this in mind, it is obvious that mud cakes made of muds with nanoparticles are formed later than those of base muds, but after forming they are less permeable and of higher quality.

Filtration through the ceramic disc which has a permeability of  $0.75 \mu\text{m}^2$  (750 mD) showed a slight increase (6%) in filtration volume after 30 minutes for mud containing  $\text{SiO}_2$  nanoparticles at a concentration of 1 wt% (mud E), while the filtration volume of mud containing  $\text{TiO}_2$  nanoparticles at a concentration of 1 wt% (mud C) was the same as those measured with the base mud (mud A). The filtration volume of mud containing  $\text{TiO}_2$  nano-

particles at a concentration of 0.5 wt% (mud B) was decreased by 17.0% in comparison to the base mud after 30 minutes of filtration, while the addition of  $\text{SiO}_2$  nanoparticles at a concentration of 0.5 wt% (mud D), the filtration volume was reduced by 10.6% in comparison to the base mud. However, observing the spurt loss, it can be seen that the amount of fluid that is lost before forming the mud cake is lower by 13.3% for muds which contain  $\text{TiO}_2$  nanoparticles at both concentrations (mud B and C) than in the case of a basic drilling mud (mud A). On the other hand, the spurt loss of muds which contain  $\text{SiO}_2$  nanoparticles at both concentrations (mud D and E) was higher than those in the base mud by up to 20%.

#### 4. Discussion of results

**Figure 6** shows a comparison of the API and HTHP filtration results for all tested drilling muds.

According to **Figure 6**, the addition of  $\text{TiO}_2$  and  $\text{SiO}_2$  nanoparticles to the base mud did not have the anticipated effect on API filtration. At HTHP conditions, tests were carried out at a differential pressure of 55 bar (800 psi) and a temperature of  $88^\circ\text{C}$  ( $192^\circ\text{F}$ ), and a greater difference in the measured filtration was observed for the different concentrations of the added nanoparticles (0.5 wt% or 1 wt%). The test was carried out with two different discs, which have a permeability of 400 and 750 mD, but in all measurements, the same trend can be observed. As can be seen in **Figure 6**, the filtration of mud with  $\text{TiO}_2$  nanoparticles added at a concentration of

0.5 wt% had the smallest filtration. Also, the filtration of mud SiO<sub>2</sub> nanoparticles added at a concentration of 0.5 wt% was less than those measured with mud that contained SiO<sub>2</sub> nanoparticles added at a concentration of 1 wt%. If these results are compared with other results presented in **Table 2**, similar trends can be seen, but to get a better insight on the influence of adding nanoparticles on filtration properties, comprehensive research is necessary. For further laboratory research, different nanoparticle types and sizes (for the same type) should be properly selected. Also, measurement should be conducted with a base drilling mud (eg. bentonite suspension) and gradually different additives should be added by following the proper procedure.

## 5. Conclusion

Based on the conducted laboratory research, it can be generally concluded that the TiO<sub>2</sub> and SiO<sub>2</sub> nanoparticles, which were used here, had an impact on filtration properties. The concentration of the added nanoparticles differently affects the filtration properties of the tested muds. Comparing both types of nanoparticles, even though both types have similar particle sizes, which was confirmed by granulometric analyses, better results were obtained by using TiO<sub>2</sub> nanoparticles. The HTHP filtration of muds was the lowest in the muds which contained TiO<sub>2</sub> and SiO<sub>2</sub> nanoparticles added at a concentration of 0.5 wt%, even lower than those measured by the addition of those nanoparticles at a concentration of 1 wt%, and it was confirmed that nanoparticles should be added at lower concentrations. Better insight will be obtained through comprehensive laboratory research which should include a wider range of different nanoparticle types and sizes added in different muds.

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## SAŽETAK

### Utjecaj dodavanja nanočestica $\text{TiO}_2$ i $\text{SiO}_2$ na filtracijska svojstva isplaka na bazi vode

Tijekom izrade kanala bušotine zbog razlike između tlaka u kanalu bušotine i slojnoga tlaka dolazi do filtracije tekuće faze iz isplake u stijene pribušotinske zone. Kako bi se smanjila količina filtrata koja prodire u stijene pribušotinske zone, potrebno je održavati određenu gustoću isplake koja neće uzrokovati tlak u bušotini veći od tlaka loma formacije. Uz to, u isplaku se preventivno dodaju materijali za očvršćavanje stijenki kanala bušotine. Čvrste čestice iz isplake, ponajprije bentonita i barita, čija se veličina čestica kreće od 0,1 do 100  $\mu\text{m}$ , odlažu se na stijenama kanala bušotine te stvaraju isplačni oblog. Tijekom izrade kanala bušotine u šejlovima koji imaju pore prosječnoga promjera od 10 do 30 nm klasični materijali ne mogu stvoriti visokokvalitetan isplačni oblog, pa tekuća faza iz isplake prodire u šejl i stvara probleme vezane uz stabilnost kanala bušotine. Zbog toga se naftna industrija okrenula primjeni isplake na bazi nafte, ali pogotovo u zadnje vrijeme zbog njezina nepovoljnog utjecaja na okoliš traže se nova rješenja koja će omogućiti primjenu isplaka na bazi vode. Stoga se u posljednje vrijeme sve više provode laboratorijska ispitivanja uporabe nanočestica koje zbog svojih malih dimenzija mogu ući u nanopore, ispuniti taj prostor i ojačati stijenke kanala bušotine, što u konačnici rezultira smanjenom filtracijom. U ovome radu prikazan je pregled prethodnih laboratorijskih istraživanja utjecaja nanočestica na filtraciju. Osim toga, ispitan je utjecaj dodavanja nanočestica  $\text{TiO}_2$  i  $\text{SiO}_2$ , u koncentraciji od 0,5 i 1 mas % u pet različitih isplaka na bazi vode, na API filtraciju te filtraciju u uvjetima visokoga tlaka i temperature (HTHP). Najbolji rezultat dobiven je dodavanjem nanočestica  $\text{TiO}_2$  u koncentraciji 0,5 mas %.

#### Ključne riječi:

nanočestice  $\text{TiO}_2$  i  $\text{SiO}_2$ , isplaka, filtracija, stabilnost kanala bušotine

#### Authors contribution

This paper is a part of the PhD research of the author **Petar Mijić** (PhD student, graduate engineer of petroleum engineering) who initialized the idea, lead the laboratory research, participated in interpretation of the laboratory results and wrote the paper. The supervisor of the PhD thesis, **Nediljka Gaurina-Međimurec** (PhD, Full Professor) provided the evaluation of the overall laboratory testing results and made a critical revision of the paper. **Borivoje Pašić** (PhD, Assistant Professor) participated in the interpretation of the results, in discussion of the results and made a critical revision of the paper. **Igor Medved** (PhD student, graduate engineer of petroleum engineering) participated in the laboratory research and in preparing a review of previous laboratory tests. All authors participated in writing the conclusion of this paper.