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Research paper

Experimental investigation of fluid loss and cake thickness control ability of zirconium (IV) oxide (Z_RO_2) nanoparticles in water based drilling mud.

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Abstract

A major technical and economical concern of the oil well drilling industry is the potential damage to productive formations because of excessive drilling fluid filtration and its multiplier effect on cake thickness. High fluid loss (high cake permeability) results in thick filter cake which reduces the effective diameter of the hole (tight holes) and causes various problems such as excessive torque when rotating the pipe, excessive drag when pulling it and high swab and surge pressures due to reduced hole diameter and differential pipe sticking due to increase in pipe contact.
It is in this light that the potential of Zirconium (IV) oxide (Z_r O_2) nanoparticles in combating excessive filtration problem in Water Based Mud was investigated. Preliminary results show that addition of 0.50g (Z_r O_2) nanoparticle concentration brought about 19.10% reduction in fluid loss and 14.29 % reduction in cake thickness for the High Temperature/ High Pressure (HPHT) filtration test at 500psi and 250 ^{O}F. Similarly, the highest reduction of 48.31% and 38.10% in fluid loss and cake thickness respectively was achieved with addition of an optimum concentration of 2.0g of (Z_r O_2) nanoparticles for the HTHP filtration test at the same temperature and pressure.

Keywords: *Cake Permeability; Fluid Loss; HTHP; Nanoparticles; Water Based Mud (WBM); Zirconium (IV) Oxide.*

1. Introduction

The Oil well drilling industry started with water base fluids in various early drilling operations principally because of its lower costs, ease of formulation and its environmental friendliness [1]. However, because of formation clays that react, swell or slough after exposure to water based mud as a result of excessive filtration which invariably lead to excessive formation damage coupled with the need to penetrate a hole with high temperature, a shift from water based mud to oil based mud began in 1960.
Similarly, [2] concluded that the excessive filtration reduces penetration rate when he observed that penetration rates (drilling rate) were found to be related qualitatively to the difference between the filtration rates measured while drilling and circulating by using four water based mud samples to drill four water based mud samples to drill four saturated sand stone formation samples during laboratory drilling tests to measure the effect of pore pressure and mud filtration on penetration rates.
Hence, the petroleum industry has spent large sums of money during drilling operation on different commercial fluid loss control agents such as Carboxyl methyl cellulose (CMC), Polyacrylate and Starch to control the fluid loss properties of drilling fluids [3].
All the above mentioned filtration control additives have limitations at high temperatures, salinity or hardness and can also increase mud viscosity. Although, Starch is relatively unaffected by water salinity or hardness and thus primarily used in muds with high salt concentration but it cannot be used in muds that are exposed to high temperatures because its thermal degradation begins at about 200^{O}F and it is also subjected to bacterial action and must be used with preservative except in saturated salt water muds or muds with a PH above 11.50 [4]. While CMC can be used at high temperature up to 300^{O}F, it tends to deflocculate clay at low concentrations and thus lowers gel strength and yield point in addition to water loss. Sodium Polyacrylate is even more temperature stable than CMC but it is extremely sensitive to calcium.
A more conventional method of reducing filtrate loss is the addition of Organophilic clay because filtrate loss decreases with increase in the concentration of solid but the cake volume increases. Hence if a drilling operator adds extra clay to a mud to reduce the filter loss, he may believe that he is also reducing cake thickness but he is actually increasing it [5]. Hence, one is faced with the dire consequences of thick filter cake on both the formation and the drilling equipment.
A Nano-based drilling fluid is a fluid that has a nanoparticle size ranging between 1-100 nanometres (nm) in size. These particles have a surrounding interfacial layer which typically consists of ions, inorganic and organic molecules. Nanoparticles can occur naturally and it can also be produced by gas condensation, attrition, chemical precipitation, ion implantation, pyrolysis and hydrothermal synthesis.
Nanoparticles are applied in drilling fluids mainly to form a thin layer of non-erodible and impermeable nanoparticles membrane around the well bore which prevents common problems such as clay swelling, spurt loss and mud loss due to circulation [6]. These nanoparticles are able to form a hydrophobic film on the head of the drill [7].

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2. Materials and method

2.1. Zirconium (IV) oxide (Z_rO_2) nanoparticle

Zirconium (IV) oxide (Z_rO_2), sometimes known as Zirconia, is a white crystalline non- flammable oxide of Zirconium. It occurs naturally with a monoclinic crystalline structure as a mineral called Baddeleyite. It can also be obtained by the reaction between of Zircon (naturally occurring Zirconium) with coke, iron borings and lime until the silica is reduced to silicon that alloys with the iron. The Zirconia is then stabilized by heating it to about 3,095 degree Fahrenheit (1700 degree Celcius) with additions of lime and magnesia totaling about five percent. Unlike other ceramic materials, Zirconium (iv) oxide (Z_rO_2) is a material with a very high resistance to crack propagation.
Zirconium oxide ceramics also have very high thermal expansion and are therefore the material of choice for joining ceramics and steel. The world largest Zircon mines are in Australia, South Africa and the United states while Baddeleyite occurs significantly in Brazil and Florida. The physical properties and particulate nature of the nanoparticle is shown in table1 while Figure 1 represents its transmission electron microscope image (TEM).

Table 1: Physical Properties and Particulate Nature of Zirconium (IV) oxide (Z_rO_2).

Form	Nano powder
Appearance	White powder
Structure	Crystalline
Particle size	48 nm
Surface area	$\geq 25\ ^{m^2}/_{g}$
Density	$5.68\ ^{g}/_{cm^3}$ at room temperature
Boiling point	4,300 ^{O}C
Melting point	2715 ^{O}C
Average molecular mass	$123.22\ ^{g}/_{mol}$
Purity	99.9%

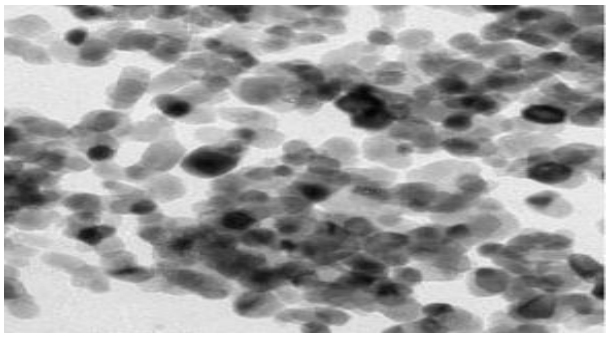


Fig. 1: TEM Image of Zirconium (IV) oxide Nanoparticle.

2.2. Experimental design

In an attempt to investigate the effect of Zirconium (IV) oxide nanoparticle on the filtration properties of water based mud, five different samples of WBMs were prepared. These samples are highlighted below.

a) Control sample of WBM with no nanoparticle
b) WBM sample with 0.5g Zirconium (iv) oxide (Z_rO_2) nano particle
c) WBM sample with 1.0g Zirconium (iv) oxide (Z_rO_2) nano particle
d) WBM sample with 1.5g Zirconium (iv) oxide (Z_rO_2) nano particle
e) WBM sample with 2.0g Zirconium (iv) oxide (Z_rO_2) nano particle

2.3. Formulation of a bentonite gel water based mud

A Bentonite-gel Water Based Mud (WBM) was prepared by carefully measuring 350ml of fresh water with the aid of measuring cylinder and poured inside the Hamilton Beach multi mixer cup shown in Figure 2 and 22.50g of bentonite was measured by using digital weighing balance and this was added to the already measured 350ml fresh water in the multi mixer cup. These two components were stirred for about ten minutes to obtain a homogenous mixture. This was followed by the addition of 20g of potassium chloride (Kcl) which helps to inhibit shale swelling. The mixture was stirred for another ten minutes and 1.50 g of caustic soda was added to the mixture to control PH. The resulting mixture was stirred followed by addition of 2.0g soda ash for the treatment of any possible calcium contamination. Finally, 140g of barite was added to the mixture as weighting agent and the entire mixture was stirred for two hours and hot rolled for about twelve hours. For samples B,C,D and E, the measured quantities of Zirconium (iv) oxide (Z_rO_2) nano particles were added prior to the final addition of weighting agent.



Fig. 2: Hamilton Beach Multi-mixer.

2.4 Static high temperature high-pressure test

The filtration properties of drilling fluids are usually evaluated and controlled by the API filter loss test [8]. The filter press (Figure 3) is used to determine the filtration rate through a standard filter paper and the rate at which the mud cake thickness increases on the standard filter paper under standard test conditions. In order to carry out this test, the heating jacket was connected to a power supply and a thermometer in a thermometer well outside the jacket. The jacket was preheated to about 10^{O}F above the test temperature of 300^{O}F and this was maintained with the aid of a thermostat. Freshly agitated mud samples were poured into the properly assembled filter press cells to about 0.50 inch from the lid in order to minimize contaminations of the filtrate produced. The top cap was then checked to ensure that the gasket was in place and well seated and the assembly was completed by tightly fixing the top cap. The whole cell assembly was placed into the frame and secured with the T –screw. A pressure of 100 psi was applied to the pressure units and the top valve stem was opened by turning it counter clockwisely to about one quarter. When the test temperature was reached, the top pressure unit was increased to 600psi and the bottom valve was opened by turning it one-quarter clockwisely to begin filtration. The filtrate was then carefully drained from the receiver when the back pressure exceeds 100psi while the standard filter paper was removed to determine its cake thickness by using a pair of Vernier calipers. This procedure was repeated for all the five samples.



Fig. 3: High Temperature High Pressure Filter Press.

2.5. Low temperature low-pressure filtration test

The low temperature low pressure filtration test is performed with the filter press shown in Figure 4 below. The mud sample to be filtered was agitated with the aid of Hamilton beach mixer and its temperature was measured with the aid of a thermometer. The mud sample was poured into the filter cell within the 0.25 inch of the zero ring groove with the standard filter paper in place. The lid of the filter press was placed between the flanges of the cell and turned clockwise until hand tight. The cell was turned over and the male cell coupling was inverted into the female filter press coupling and was turned in either direction to engage the duo. A clean-dried graduated cylinder was placed under the filtrate exit tube to receive the filtrate. A pressure of 100 psi was applied into the system by opening the inlet valve. The filtrate volume was recorded at the desired time intervals of 5,10,15,20,25 and 30 minutes. The valve was closed after recording the volume of the filtrate at the desired time intervals.



Fig. 4: Low Temperature Low Pressure Filter Press.

3. Result and discussion

a) Effect of Zirconium (IV) oxide Nanoparticle on Fluid Loss.
The addition of Zirconium (iv) oxide nanoparticle significantly reduced the fluid loss as shown in Tables 2,3,4 and 5 and Figures 5,6,7 and 8 for the low temperature low pressure filter press. The HTHP filtration process also showed a reasonable reduction in fluid as seen in Table 6 and Figure 9 respectively. The observed reduction in fluid loss is as a result of the fact that the dispersed nanoparticles in the mud system act as bridging agent and sealed the permeable filter cake at Nano-scale producing a very firm and crack free filter cake surface [9]. Similar results have been reported [10-12] on the potential of reduction of fluid loss by addition of different types of nanoparticle to Water Based Muds. This lower amount of fluid loss results in minimum swelling of clay and minimum damage to productive formation.

Table 2: LTLP Filtrate Loss in Ml at 100 Psi and 27^{O}C

Time(Min)	SAM-PLE A	SAM-PLE B	SAM-PLE C	SAM-PLE D	SAM-PLE E
5	2.60	2.20	1.90	1.65	1.50
10	3.50	3.10	2.70	2.50	2.20
15	4.20	3.80	3.30	3.00	2.80
20	4.90	4.40	3.90	3.50	3.10
25	5.40	5.00	4.50	4.00	3.60
30	5.90	5.50	5.00	4.30	3.90

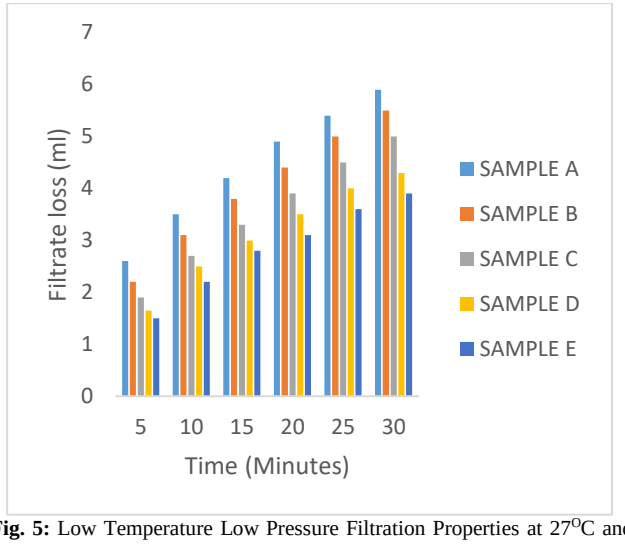


Fig. 5: Low Temperature Low Pressure Filtration Properties at 27^{O}C and 100psi.

Table 3: Filtrate Loss in Ml at 100psi and 50^{O}C

Time(Min)	SAM-PLE A	SAM-PLE B	SAM-PLE C	SAM-PLE D	SAM-PLE E
5	3.40	2.80	2.50	2.10	1.80
10	4.50	3.70	3.40	2.90	2.50
15	5.60	4.60	4.30	3.50	3.10
20	6.50	5.40	4.90	4.20	3.60
25	7.20	6.10	5.60	4.80	4.10
30	7.90	6.80	6.10	5.40	4.60

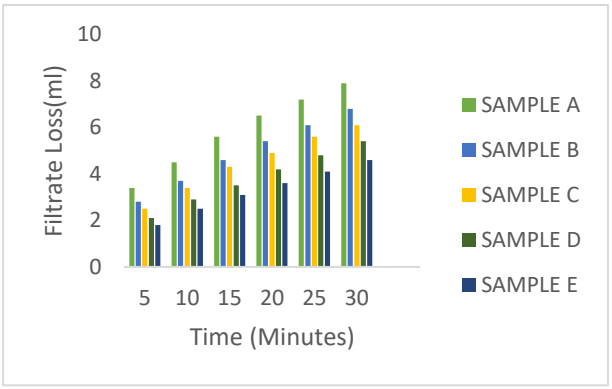


Fig. 6: Graphical Representation of Filtration Properties at 50^{O}C and 100psi.

Table 4: Filtrate Loss in Ml at 100 Psi and 75^{O}C

Time(Min)	SAM-PLE A	SAM-PLE B	SAM-PLE C	SAM-PLE D	SAM-PLE E
5	4.00	3.40	3.00	2.60	2.20
10	5.30	4.50	3.90	3.40	2.90
15	6.50	5.50	4.70	4.10	3.40
20	7.30	6.40	5.40	4.80	4.00
25	8.10	7.20	6.10	5.40	4.60
30	8.90	8.00	6.90	6.00	5.10

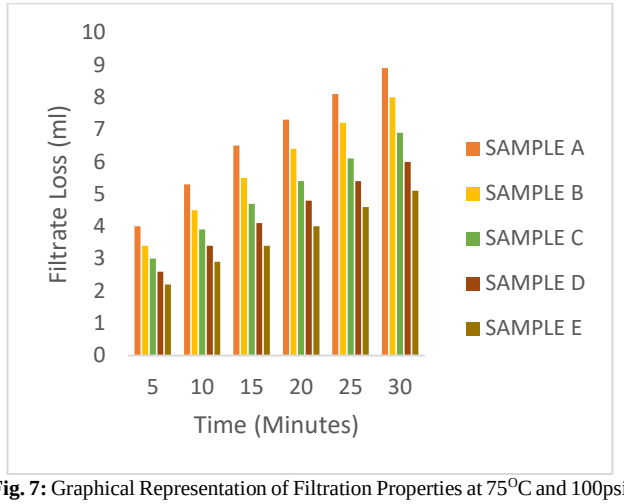


Fig. 7: Graphical Representation of Filtration Properties at 75^{O}C and 100psi.

Table 5: Filtrate Loss in Ml at 100 Psi and 100^{O}C

Time(Min)	SAM-PLE A	SAM-PLE B	SAM-PLE C	SAM-PLE D	SAM-PLE E
5	4.60	3.90	3.50	3.10	2.60
10	6.20	5.40	4.60	3.90	3.30
15	7.40	6.60	5.40	4.80	3.90
20	8.20	7.50	6.20	5.50	4.50
25	9.00	8.10	7.00	6.10	5.20
30	9.80	8.90	7.70	6.80	5.90

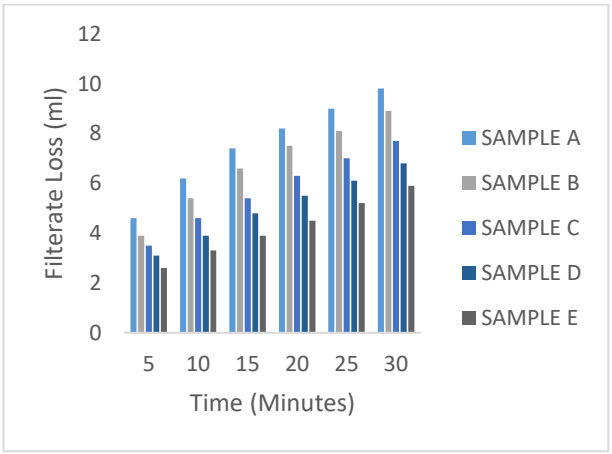


Fig. 8: Graphical Representation of Filtration Properties at 100 Psi and 100^{O}C.

HTHP FILTERATION PROPERTIES

Table 6: Hpht Static Filterate Volume AT 500 Psi and 250OF

MUD SAMPLE	FILTERATE VOLUME (ml)
A	17.80
B	14.40
C	12.60
D	10.80
E	9.20

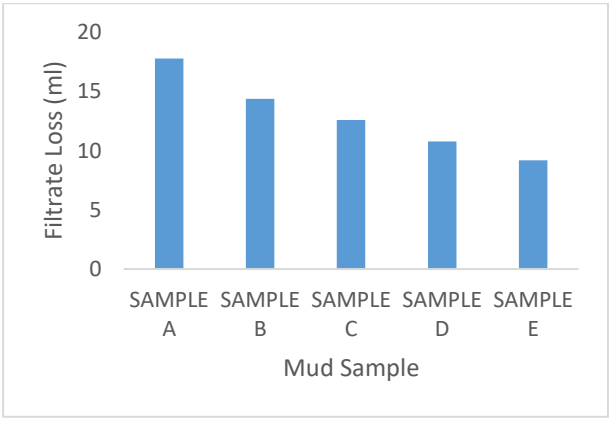


Fig. 9: HPHT Static Filtrate Volume at 500 PSI and 250^{O}F.

b) Effect of Zirconium (IV) oxide Nanoparticle on Cake Thickness.

Cake permeability provides useful information on the electrochemical conditions prevailing in the mud and it is a fundamental parameter that controls both static and dynamic filtration. The lower amount of fluid loss results in a thin filter cake on addition of Zirconium (iv) oxide nanoparticle compared with the mud sample A with no Nanoparticles. The decrease in cake permeability is attributed to a considerable decrease in mean particle diameter and an increase in the width of particle size range. A thin filter cake has a potential for reducing the differential pressure sticking problem and formation damage while drilling [13]. Muds must be treated to keep cake permeabilities as low as possible in order to maintain a stable borehole and to minimize filtrate invasion of and damage to potentially productive zone or horizons. The Filter cake thickness increases with temperature as it can be observed from tables 7,8,9,10, 11 and Figures 10,11,12,13 and 14.

Table 7: Cake Thickness for LTLP Filtrate at 100 Psi and 27^{O}C

MUD SAMPLE	Cake Thickness (mm)
A	0.40
B	0.34
C	0.28
D	0.25
E	0.20

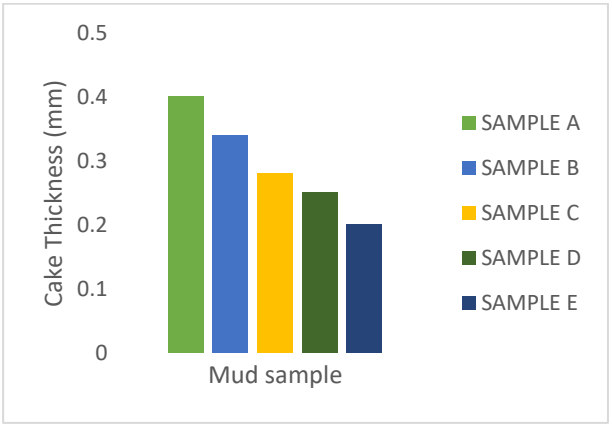


Fig. 10: Cake Thickness for LTLP Filtrate at 100 Psi and 27^{O}C.

Table 8: Cake Thickness for Filtrate at 100 Psi and 50^{O}C

MUD SAMPLE	Cake Thickness (mm)
A	0.48
B	0.45
C	0.40
D	0.35
E	0.28

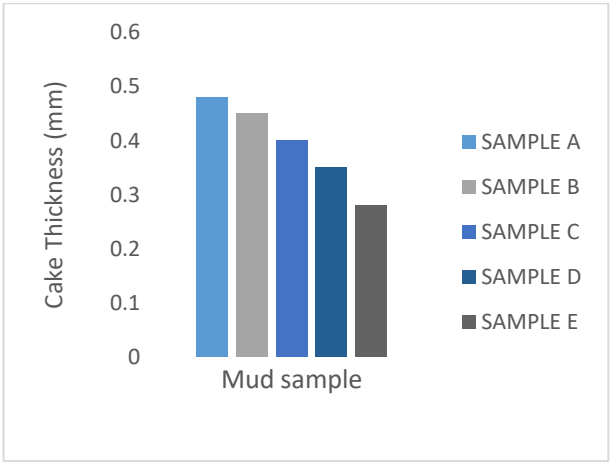


Fig. 11: Cake Thickness for Filtrate at 100 Psi and 50^{O}C.

Table 9: Cake Thickness for Filtrate at 100 Psi and 75^{O}C

MUD SAMPLE	Cake Thickness (mm)
A	0.54
B	0.50
C	0.46
D	0.42
E	0.36

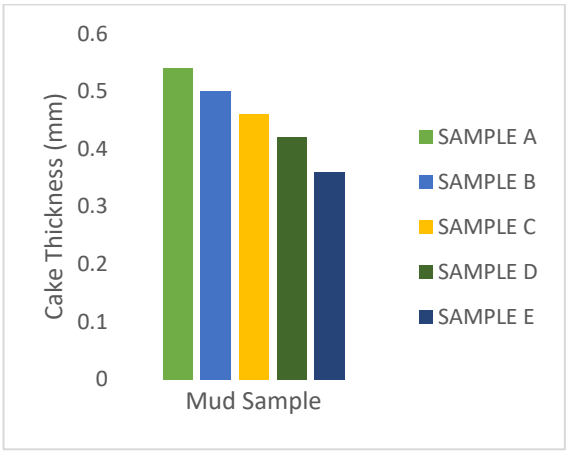


Fig. 12: Cake Thickness for Filtrate at 100 PSI and 75^{O}C.

Table 10: Cake Thickness for LTLP Filtrate at 100 Psi and 100^{O}C

MUD SAMPLE	Cake Thickness (mm)
A	0.60
B	0.55
C	0.49
D	0.45
E	0.40

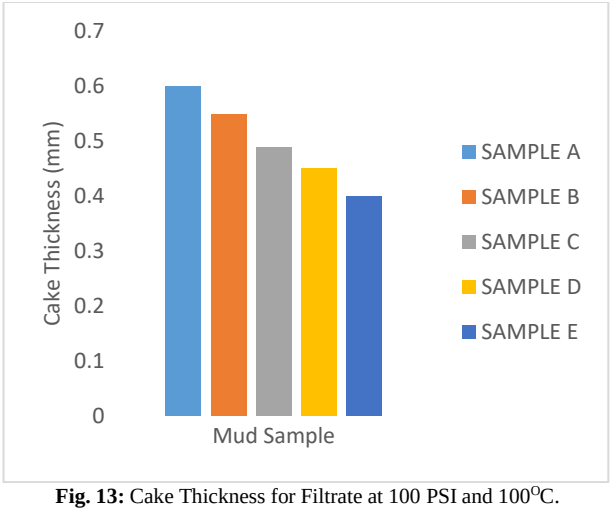


Fig. 13: Cake Thickness for Filtrate at 100 PSI and 100^{O}C.

Table 11: Cake Thickness for HTHP Filtrate at 500 Psi and 250^{O}F

MUD SAMPLE	Cake Thickness (mm)
A	0.84
B	0.72
C	0.65
D	0.58
E	0.52

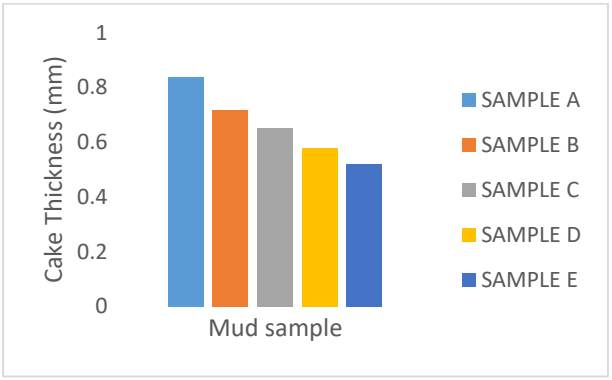


Fig. 14: Cake Thickness for HTHP Filtrate at 500 Psi and 250^{O}F.

a) Relationship between Filtrate Volume and Time.

The filtrate volume increases with increase in time as it can be seen in Tables 2,3,4,5 and 6 at constant temperature and pressure.

b) Effect of Temperature on Filtrate Volume.

The filtrate volume increased with temperature at constant pressure as observed in Figures 5,6,7,8 and 9. This phenomenon can be attributed to reduction in filtrate viscosity, changes in electrochemical equilibrium, which governs the degree of flocculation and aggregation as well the chemical degradation of one or more components of the mud. In addition, temperature is a measure of the average kinetic energy of a molecule, hence at higher temperature; the mobile phase of the mud developed more kinetic energy to move through the porous filter paper.

c) Effect of Pressure on Filtrate volume.

An increase in filtrate volume was observed with all the mud samples when the pressure was increased at constant temperature. The reason for this behavior is that bentonite is almost entirely composed of finely divided platelets of montmorillonite, which tend to align parallel to the substrate with increase in pressure.

4. Conclusion

An investigation has been conducted to critically examine the effect of Zirconium (IV) oxide (Z_rO_2) on the filtration properties of Water Based Mud via experimentation with five different WBM samples. The following inferences can be drawn from the observation.

- Zirconium (IV) oxide nanoparticle can reliably reduce filtrate loss and cake thickness when added to WBMs in controlled amount.
- The fluid loss and cake thickness reduces with an increase in nanoparticle concentration but increases with pressure and time.
- Addition of 2.0g of the nanoparticle brought about 33.89% in fluid loss and 50% reduction in cake thickness for low temperature low-pressure static filtration test at 100psi and 27^{O}C.
- A maximum reduction of 48.31% in fluid loss and 38.10% in cake thickness was achieved with nanoparticle concentration of 2.0g for HTHP static filtration test at 100psi and 250^{O}F.

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