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Abstract

Energy industries have been taking advantages from the recent developments of nanotechnology. Several revolutionary changes can be made in drilling industry with the help of nanotechnology. It has a capability to produce such nanomaterials that can bring benefit to the industry in various manners such as improving the quality of mud cake, decreasing the frictional resistance in the well, minimizing the risk of pipe sticking, establishing borehole stability, preventing reservoir from formation damage, and augmenting the recovery of oil and gas. In the field of drilling fluids, researchers have tested the application of nanoparticles and have concluded with very promising results in terms of reduction in torque and drag, stabilizing the well bore, controlling fluid loss and improving hole cleaning efficiency.

Use of the nanoparticles in reducing friction and improving lubrication effect has already been recognized in different industries. Drilling industry has moved towards exploiting reservoir in more economical ways such as drilling an extended reach well. These wells are quite challenging when it comes to solving down hole drilling problems such as high torque and drag. Lubrication efficiency of drilling fluids can play a decisive role in solving torque and drag problem. Although oil based drilling fluid offers reduced friction factor in the wellbore, environmental concerns and high expenditures can limit its usage. An alternate solution is to make the most of better lubrication efficiency of the nanoparticles by adding them in drilling fluid particularly to water base drilling fluids. This addition also has its impact on the rheological properties of drilling fluid which can affect its other function.

By keeping this agenda in mind, different nanoparticles were added to drilling fluids at different concentrations and the impact on friction factor and viscosity was studied through tribometer and rheometer respectively. Graphene, carbon nanotubes, silica, alumina, cobalt and nickel nanoparticles were selected as potential particles and their effects were studied in three different base fluids. Temperature influence on tribology and rheology were also made part of the study. The reliability of test results was established by using Modular Compact Rheometer (MCR) for rheological measurements and Pin-on-Disc Tribometer for friction factor measurements. In addition to this, the impact of nanoparticles' addition on density and pH of drilling fluid was also evaluated and the results were used to ponder on the relationship between different properties of drilling fluid.

Evaluation of viscosity and friction factor led to selection of the best nano drilling fluids which were further used in case study to develop temperature, viscosity, friction factor and torque and drag profile using software technology. The comparison of results between conventional drilling fluid and nano drilling fluids proves that nano drilling fluid can deliver better performance in terms of better bit cooling, enhanced viscous behavior, low friction factors, and reduced torque and drag.

Table of Contents

A	cknow	ledge	ementiii
A	bstrac	t	v
L	ist of F	ligure	esxi
L	ist of T	Tables	sxiii
N	omenc	latur	exv
A	bbrevi	ation	sxvii
1	Inti	roduc	ction1
2	Dri	lling	Fluid and Nanoparticles
	2.1	Intro	oduction to Drilling Fluid
	2.2	Basi	c Functions of Drilling Fluid3
	2.3	Туре	es of Drilling Fluids
	2.3.	1	Water Based Fluids4
	2.3.	2	Drill-In Fluids (DIFs)
	2.3.	3	Oil Based Fluids
	2.3.	4	Synthetic-Based Drilling Fluids5
	2.3.	5	All-Oil Fluids
	2.3.	6	Pneumatic-Drilling Fluids6
	2.4	Nan	otechnology6
	2.4.	1	Definition of Nanoparticle6
	2.4.	2	Properties of Nanoparticles7
	2.5	Nan	otechnology and Petroleum Industry7
	2.5.	1	Exploration
	2.5.	2	Drilling and Completion8
	2.5.	3	Production8
	2.5.	4	Reservoir Characterization9
	2.5.	5	Refinery and Processing9
	2.6	Nan	o Drilling Fluid Research – A Brief Summary9
	2.7	Com	nparison between Conventional and Nano Drilling Fluid11
	2.8	Pote	ential Nanoparticles
	2.9	Drill	ing Fluid Rheology17

	2.9.3	1	Newtonian vs. Non-Newtonian Fluids	. 17
	2.9.2	2	Rheological Models	. 19
	2.10	Dril	ling Fluid Tribology	.21
	2.10	.1	Wear Phenomenon	.21
	2.10	.2	Friction	.24
	2.10	.3	Factor affecting Tribological Behavior	. 25
3	Exp	erin	nental Setup	.27
	3.1	Rhe	ometer	. 27
	3.2	Trib	ometer	.31
	3.3	Der	sity Meter	. 33
	3.4	рН	Meter	.34
4	Exp	erin	nental Procedure	.35
	4.1	Nar	o-Based Fluid Preparation	.35
	4.1.3	1	Base fluids Preparation	. 35
	4.1.2	2	Nanoparticle Addition	. 37
	4.2	Visc	osity Measurements	. 38
	4.3	Fric	tion Factor Measurement	.40
	4.4	Der	sity Measurement	.42
	4.5	рH	Measurement	.43
5	Res	ults	and Discussions	.44
	5.1	MC	R Measurements	.44
	5.1.3	1	Pure Water	.44
	5.1.2	2	Base Fluid 1	.48
	5.1.3	3	Base Fluid 2	.63
	5.2	Trib	ometer Measurements	.78
	5.2.2	1	Pure Water	.78
	5.2.2	2	Base Fluid 1	. 79
	5.2.3	3	Base Fluid 2	.81
	5.3	Der	sity Measurements	.83
	5.3.3	1	Pure Water	.83
	5.3.2	2	Base Fluid 1	.84
	5.3.3	3	Base Fluid 2	. 85

5	5.4	pH Measurements
	5.4.1	Pure Water
	5.4.2	Base Fluid 1
	5.4.3	Base Fluid 288
6	Mod	leling Work
6	5.1	Introduction
6	5.2	Selection of Base Fluid and Nanoparticles89
6	5.3	Case Study
6	5.4	Temperature Model90
6	5.5	Viscosity Model
6	5.6	Friction Factor Model96
(5.7	Torque and Drag Model98
7	Con	clusions and Recommendations103
7	7.1	Conclusions
7	7.2	Recommendations for Future Work104
Re	ferenc	es105

List of Figures

Figure 2.2: Graphene [41] 14 Figure 2.3: Image of Nanosilica obtained from Scanning Electron Microscopy (SEM) [43] 14 Figure 2.4: SWCNT [44] 15 Figure 2.5: MWCNT [44] 15 Figure 2.6: Nanoalumina [45] 16 Figure 2.8: Nickel Nanopowder [47] 16 Figure 2.9: Nate Experiment [48] 18 Figure 2.9: Nate Experiment [48] 18 Figure 2.10: Fluid Types [49] 19 Figure 2.11: Rheological Models [50] 19 Figure 2.12: Abrasive Wear [51] 22 Figure 2.13: Adhesive Wear [51] 23 Figure 2.16: Friction as function of Applied Force [53] 24 Figure 2.17: Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.1: Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.1: Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate at the Bottom, 29 Figure 3.3: Modular Compact Rheometer 29 Figure 3.4: Pressure Cell Assembly [59] 30 Figure 3.5: Double Gap Measuring System [59] 32 Figure 3.6: Forces in Tribometer [60] 32 Figure 3.7: Tribometer 32 <	Figure 2.1 : Surface Area Modification using Nanoparticles [14]	6
Figure 2.3 : Image of Nanosilica obtained from Scanning Electron Microscopy (SEM) [43]	Figure 2.2 : Graphene [41]	14
Figure 2.4 : SWCNT [44] 15 Figure 2.5 : MWCNT [44] 15 Figure 2.5 : NWCNT [44] 16 Figure 2.7 : Cobalt nanopowder [47] 16 Figure 2.7 : Cobalt nanopowder [47] 17 Figure 2.9 : Plate Experiment [48] 18 Figure 2.9 : Plate Experiment [48] 19 Figure 2.1 : Rheological Models [50] 19 Figure 2.12 : Abrasive Wear [51] 22 Figure 2.13 : Adhesive Wear [51] 23 Figure 2.14 : Fatigue Wear [51] 23 Figure 2.15 : Erosive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylinders [59] 30 Figure 3.3 : Modular Compact Rheometer 28 Figure 3.4 : Pressure Cell Assembly (59) 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32	Figure 2.3 : Image of Nanosilica obtained from Scanning Electron Microscopy (SEM) [43]	14
Figure 2.5 : MWCNT [44] 15 Figure 2.5 : Nanoalumina [45] 16 Figure 2.7 : Cobalt nanopowder [47] 16 Figure 2.8 : Nickel Nanopowder [47] 17 Figure 2.9 : Plate Experiment [48] 18 Figure 2.9 : Plate Experiment [48] 19 Figure 2.10 : Fluid Types [49] 19 Figure 2.11 : Rubeological Models [50] 19 Figure 2.12 : Abrasive Wear [51] 22 Figure 2.13 : Adhesive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 23 Figure 2.17 : Extended Reach Well [55] 25 Figure 3.17 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 29 Figure 3.4 : Pressure Cell Assembly [59] 30 31 Figure 3.5 : Double Gap Measuring System [59] 32 32 Figure 3.8 : Density Meter 33 32 Figure 4.1 : Precision Balance 36 36 Figure 4.2 : Mixer 36 34 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.4 : Prescion Bala	Figure 2.4 : SWCNT [44]	15
Figure 2.6 : Nanoalumina [45] 16 Figure 2.7 : Cobalt nanopowder [47] 16 Figure 2.8 : Nickel Nanopowder [47] 17 Figure 2.9 : Plate Experiment [48] 18 Figure 2.10 : Fluid Types [49] 19 Figure 2.11 : Rheological Models [50] 19 Figure 2.12 : Abrasive Wear [51] 22 Figure 2.13 : Adhesive Wear [51] 22 Figure 2.14 : Fatigue Wear [51] 22 Figure 2.15 : Erosive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 2.17 Extended Reach Well [55] 25 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.1 : Schematic Diagram (a) Concentre Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.1 : Schematic Diagram (a) Concenter 29 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 16 26 (c) Hollow Cavity at the Bottom to Trap Aris [75] 30 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Trb	Figure 2.5 : MWCNT [44]	15
Figure 2.7 : Cobalt nanopowder [47] 16 Figure 2.8 : Nickel Nanopowder [47] 17 Figure 2.9 : Plate Experiment [48] 18 Figure 2.10 : Fluid Types [49] 19 Figure 2.11 : Rheological Models [50] 19 Figure 2.12 : Abrasive Wear [51] 22 Figure 2.13 : Adhesive Wear [51] 23 Figure 2.14 : Faigue Wear [51] 23 Figure 2.15 : Erosive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 2.17 Extended Reach Well [55] 25 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 29 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.5 : Double Gap Measuring System [59] 30 Figure 3.7 : Tribometer 32 Figure 3.7 : Tribometer 32 Figure 3.8 : Density Meter 33 Figure 3.9 : Oscillation of Leaf Spring System 34 Figure 4.1 : Precision Balance 36	Figure 2.6 : Nanoalumina [45]	16
Figure 2.8 : Nickel Nanopowder [47] 17 Figure 2.9 : Plate Experiment [48] 18 Figure 2.10 : Fluid Types [49] 19 Figure 2.11 : Rheological Models [50] 19 Figure 2.12 : Abrasive Wear [51] 22 Figure 2.13 : Adhesive Wear [51] 22 Figure 2.15 : Erosive Wear [51] 23 Figure 2.15 : Erosive Wear [51] 23 Figure 2.17 Extended Reach Well [55] 24 Figure 2.17 Extended Reach Well [55] 25 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 28 (c) Hollow Cavity at the Bottom to Trap Air [57] 29 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.5 : Double Gap Measuring System [59] 30 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 3.10 : pH Meter 34 Figure 3.10 : pH Meter 36 Figure 3.10 : pH Meter 36 Figure 3.10 : pH Meter 36 Figure 4.1 : Pressure Cell with Assembled Components. 39 <td>Figure 2.7 : Cobalt nanopowder [47]</td> <td>16</td>	Figure 2.7 : Cobalt nanopowder [47]	16
Figure 2.9 : Plate Experiment [48] 18 Figure 2.10 : Fluid Types [49] 19 Figure 2.11 : Rheological Models [50] 19 Figure 2.12 : Abrasive Wear [51] 22 Figure 2.13 : Adhesive Wear [51] 22 Figure 2.14 : Fatigue Wear [51] 23 Figure 2.15 : Erosive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 28 (c) Hollow Cavity at the Bottom to Trap Air [57] 28 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 33 Figure 4.8 : Density Meter 36 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer. 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.4 : Pressure Cell with Assembled Components 39 <	Figure 2.8 : Nickel Nanopowder [47]	17
Figure 2.10 : Fluid Types [49] 19 Figure 2.11 : Rheological Models [50] 19 Figure 2.12 : Abrasive Wear [51] 22 Figure 2.13 : Adhesive Wear [51] 22 Figure 2.14 : Fatigue Wear [51] 23 Figure 2.15 : Erosive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 2.17 Extended Reach Well [55] 25 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 28 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 28 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, (c) Hollow Cavity at the Bottom to Trap Air [57] 28 Figure 3.3 : Modular Compact Rheometer 29 30 Figure 3.4 : Pressure Cell Assembly [59] 30 30 Figure 3.5 : Double Gap Measuring System [59] 31 32 Figure 3.10 : pH Meter 32 32 Figure 3.10 : pH Meter 32 Figure	Figure 2.9 : Plate Experiment [48]	18
Figure 2.11 : Rheological Models [50] 19 Figure 2.12 : Abrasive Wear [51] 22 Figure 2.13 : Adhesive Wear [51] 23 Figure 2.14 : Fatigue Wear [51] 23 Figure 2.15 : Erosive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 2.17 Extended Reach Well [55] 25 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 28 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.7 : Tribometer 32 Figure 3.9 : Oscillation of Leaf Spring System 34 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell with Assembled Components 39 Figure 4.4 : Pressure Cell with Assembled Components 38 Figure 4.5 : MCR during Measurement 40 Figure 4.5 : Pressure Cell with Assembled Components 39 Figure 4.6 : Heating Element in Test Fluid 41	Figure 2.10 : Fluid Types [49]	19
Figure 2.12 : Abrasive Wear [51] 22 Figure 2.13 : Adhesive Wear [51] 22 Figure 2.14 : Fatigue Wear [51] 23 Figure 2.15 : Erosive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 2.17 Extended Reach Well [55] 25 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 28 (c) Hollow Cavity at the Bottom to Trap Air [57] 28 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.1 : Pressure Cell Assembly [59] 30 Figure 3.10 : pH Meter 33 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.1 : Precision Balance 39 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell with Assembled Components 39 Fi	Figure 2.11 : Rheological Models [50]	19
Figure 2.13 : Adhesive Wear [51] 22 Figure 2.14 : Fatigue Wear [51] 23 Figure 2.15 : Brosive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 2.17 : Extended Reach Well [55] 25 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 28 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 28 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 3.8 : Density Meter 33 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.3 : Pressure Cell Components and other Accessories 39 Figure 4.3 : MCR during Measurement 40 Figure 4.3 : MCR during Kasembled Components 39 Fig	Figure 2.12 : Abrasive Wear [51]	22
Figure 2.14 : Fatigue Wear [51] 23 Figure 2.15 : Erosive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 2.17 Extended Reach Well [55] 25 Figure 2.17 Extended Reach Well [55] 25 Figure 2.17 Extended Reach Well [55] 25 Figure 2.17 Extended Reach Well [55] 27 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57]. 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 28 (c) Hollow Cavity at the Bottom to Trap Air [57] 28 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 4.1 : Precision Balance 34 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.4 : Pressure Cell with Assembled Components 39 Figure 4.5 : MCR during Measurement 40 <td>Figure 2.13 : Adhesive Wear [51]</td> <td>22</td>	Figure 2.13 : Adhesive Wear [51]	22
Figure 2.15 : Erosive Wear [51] 23 Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 2.17 Extended Reach Well [55] 25 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 28 (c) Hollow Cavity at the Bottom to Trap Air [57] 28 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 3.8 : Density Meter 33 Figure 3.1 : Prection Balance 36 Figure 4.1 : Prection Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.5 : MCR during Measurement 40 Figure 4.5 : MCR during Measurement 40 Figure 4.7 : Tribometer During Experiment 42 Figure 4.8 : Density Meter during Experiment 43 Figure 4.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C	Figure 2.14 : Fatigue Wear [51]	23
Figure 2.16 : Friction as function of Applied Force [53] 24 Figure 2.17 Extended Reach Well [55] 25 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 28 (c) Hollow Cavity at the Bottom to Trap Air [57] 28 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 3.8 : Density Meter 33 Figure 3.9 : Oscillation of Leaf Spring System 34 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.5 : MCR during Measurement 40 Figure 4.5 : MCR during Experiment 43 Figure 4.7 : Tribometer During Experiment 43 Figure 4.8 : Density Meter during Experiment 43 Figure 4.1 : Pressure Cell with Assembled Components 39 Figure 4.2 : Mixer 40	Figure 2.15 : Erosive Wear [51]	23
Figure 2.17 Extended Reach Well [55] 25 Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57] 27 Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, 29 (c) Hollow Cavity at the Bottom to Trap Air [57] 28 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 3.9 : Oscillation of Leaf Spring System 34 Figure 4.1 : Precision Balance 36 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.7 : Tribometer During Experiment 40 Figure 4.7 : Tribometer During Experiment 42 Figure 4.7 : Tribometer During Experiment 42 Figure 4.7 : Tribometer During Experiment 43 Figure 4.8 : Density Meter during Experiment 42 Figure 4.9 : Electrode of pH Meter in Nano Drilling Fluid 41 Figure 5.1 : Effect of 0.6 weight%	Figure 2.16 : Friction as function of Applied Force [53]	24
Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57]	Figure 2.17 Extended Reach Well [55]	25
Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, (c) Hollow Cavity at the Bottom to Trap Air [57] 28 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 3.8 : Density Meter 33 Figure 3.9 : Oscillation of Leaf Spring System 34 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.4 : Pressure Cell with Assembled Components 39 Figure 4.5 : MCR during Measurement 40 Figure 4.7 : Tribometer During Experiment 42 Figure 4.8 : Density Meter during Experiment 42 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 47 Figure 5.2 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 60°C (Bottom) 47 Figure 5.4 : Effect of 0	Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57]	27
(c) Hollow Cavity at the Bottom to Trap Air [57] 28 Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 3.7 : Tribometer 32 Figure 3.7 : Tribometer 32 Figure 3.8 : Density Meter 33 Figure 3.9 : Oscillation of Leaf Spring System 34 Figure 3.10 : pH Meter 34 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.5 : MCR during Measurement 40 Figure 4.7 : Tribometer During Experiment 42 Figure 4.7 : Tribometer During Experiment 42 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 46 Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 40 Figure 5.4 : Effect of 0.2 weight% Nanoparticles Addi	Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom,	
Figure 3.3 : Modular Compact Rheometer 29 Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 3.8 : Density Meter 33 Figure 3.9 : Oscillation of Leaf Spring System 34 Figure 3.10 : pH Meter 34 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.4 : Pressure Cell with Assembled Components 39 Figure 4.5 : MCR during Measurement 40 Figure 4.6 : Heating Element in Test Fluid 41 Figure 4.7 : Tribometer During Experiment 42 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 43 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) 47 Figure 5.4 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 50	(c) Hollow Cavity at the Bottom to Trap Air [57]	28
Figure 3.4 : Pressure Cell Assembly [59] 30 Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 3.8 : Density Meter 33 Figure 3.9 : Oscillation of Leaf Spring System 34 Figure 3.10 : pH Meter 34 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.4 : Pressure Cell with Assembled Components 39 Figure 4.5 : MCR during Measurement 40 Figure 4.6 : Heating Element in Test Fluid 41 Figure 4.7 : Tribometer During Experiment 42 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) 47 Figure 5.4 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 50	Figure 3.3 : Modular Compact Rheometer	29
Figure 3.5 : Double Gap Measuring System [59] 31 Figure 3.6 : Forces in Tribometer [60] 32 Figure 3.7 : Tribometer 32 Figure 3.8 : Density Meter 33 Figure 3.9 : Oscillation of Leaf Spring System 34 Figure 3.10 : pH Meter 34 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.3 : Pressure Cell with Assembled Components 39 Figure 4.5 : MCR during Measurement 40 Figure 4.6 : Heating Element in Test Fluid 41 Figure 4.8 : Density Meter during Experiment 42 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 46 Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 40 Figure 5.4 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 40 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 40 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 10	Figure 3.4 : Pressure Cell Assembly [59]	30
Figure 3.6 : Forces in Tribometer [60]32Figure 3.7 : Tribometer32Figure 3.7 : Tribometer32Figure 3.8 : Density Meter33Figure 3.9 : Oscillation of Leaf Spring System34Figure 3.10 : pH Meter34Figure 4.1 : Precision Balance36Figure 4.2 : Mixer36Figure 4.3 : Pressure Cell Components and other Accessories38Figure 4.4 : Pressure Cell with Assembled Components39Figure 4.5 : MCR during Measurement40Figure 4.6 : Heating Element in Test Fluid41Figure 4.7 : Tribometer During Experiment42Figure 4.8 : Density Meter during Experiment43Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom)47Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)49Figure 5.4 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C50	Figure 3.5 : Double Gap Measuring System [59]	31
Figure 3.7 : Tribometer 32 Figure 3.8 : Density Meter 33 Figure 3.9 : Oscillation of Leaf Spring System 34 Figure 3.10 : pH Meter 34 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.4 : Pressure Cell with Assembled Components 39 Figure 4.5 : MCR during Measurement 40 Figure 4.6 : Heating Element in Test Fluid 41 Figure 4.7 : Tribometer During Experiment 42 Figure 4.8 : Density Meter during Experiment 43 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 46 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 140°C 40 Figure 5.4 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 40	Figure 3.6 : Forces in Tribometer [60]	32
Figure 3.8 : Density Meter33Figure 3.9 : Oscillation of Leaf Spring System34Figure 3.10 : pH Meter34Figure 4.1 : Precision Balance36Figure 4.2 : Mixer36Figure 4.3 : Pressure Cell Components and other Accessories38Figure 4.4 : Pressure Cell with Assembled Components39Figure 4.5 : MCR during Measurement40Figure 4.6 : Heating Element in Test Fluid41Figure 4.7 : Tribometer During Experiment42Figure 4.8 : Density Meter during Experiment43Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom)46Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)47Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C50	Figure 3.7 : Tribometer	32
Figure 3.9 : Oscillation of Leaf Spring System34Figure 3.10 : pH Meter34Figure 3.10 : pH Meter36Figure 4.1 : Precision Balance36Figure 4.2 : Mixer36Figure 4.3 : Pressure Cell Components and other Accessories38Figure 4.4 : Pressure Cell with Assembled Components39Figure 4.5 : MCR during Measurement40Figure 4.6 : Heating Element in Test Fluid41Figure 4.7 : Tribometer During Experiment42Figure 4.8 : Density Meter during Experiment43Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom)46Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C47Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C50	Figure 3.8 : Density Meter	33
Figure 3.10 : pH Meter 34 Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.4 : Pressure Cell with Assembled Components 39 Figure 4.5 : MCR during Measurement 40 Figure 4.6 : Heating Element in Test Fluid 41 Figure 4.7 : Tribometer During Experiment 42 Figure 4.8 : Density Meter during Experiment 43 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 46 Figure 5.2 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) 47 Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 50	Figure 3.9 : Oscillation of Leaf Spring System	34
Figure 4.1 : Precision Balance 36 Figure 4.2 : Mixer 36 Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.3 : Pressure Cell with Assembled Components 39 Figure 4.4 : Pressure Cell with Assembled Components 39 Figure 4.5 : MCR during Measurement 40 Figure 4.6 : Heating Element in Test Fluid 41 Figure 4.7 : Tribometer During Experiment 42 Figure 4.8 : Density Meter during Experiment 43 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 46 Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 140°C 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) 49 Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 50 Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 50	Figure 3.10 : pH Meter	34
Figure 4.2 : Mixer.36Figure 4.3 : Pressure Cell Components and other Accessories38Figure 4.3 : Pressure Cell with Assembled Components.39Figure 4.4 : Pressure Cell with Assembled Components.39Figure 4.5 : MCR during Measurement40Figure 4.6 : Heating Element in Test Fluid41Figure 4.7 : Tribometer During Experiment42Figure 4.8 : Density Meter during Experiment43Figure 5.1 : Effect of pH Meter in Nano Drilling Fluid43Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom)47Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 140°C49Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C50	Figure 4.1 : Precision Balance	36
Figure 4.3 : Pressure Cell Components and other Accessories 38 Figure 4.4 : Pressure Cell with Assembled Components. 39 Figure 4.5 : MCR during Measurement 40 Figure 4.6 : Heating Element in Test Fluid 41 Figure 4.7 : Tribometer During Experiment 42 Figure 4.8 : Density Meter during Experiment 43 Figure 4.9 : Electrode of pH Meter in Nano Drilling Fluid 43 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom)46 Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 60°C (Bottom)47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)49 Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom) 50	Figure 4.2 : Mixer	36
Figure 4.4 : Pressure Cell with Assembled Components.39Figure 4.5 : MCR during Measurement40Figure 4.6 : Heating Element in Test Fluid41Figure 4.7 : Tribometer During Experiment42Figure 4.8 : Density Meter during Experiment43Figure 5.1 : Effect of pH Meter in Nano Drilling Fluid43Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom)46Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 60°C (Bottom)47Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)49Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C50	Figure 4.3 : Pressure Cell Components and other Accessories	38
Figure 4.5 : MCR during Measurement 40 Figure 4.6 : Heating Element in Test Fluid 41 Figure 4.7 : Tribometer During Experiment 42 Figure 4.8 : Density Meter during Experiment 43 Figure 5.1 : Effect of pH Meter in Nano Drilling Fluid 43 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 46 Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 140°C 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) 49 Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 50	Figure 4.4 : Pressure Cell with Assembled Components	39
Figure 4.6 : Heating Element in Test Fluid 41 Figure 4.7 : Tribometer During Experiment 42 Figure 4.8 : Density Meter during Experiment 43 Figure 4.9 : Electrode of pH Meter in Nano Drilling Fluid 43 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom)46 46 Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 140°C 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)49 49 Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 50 Kottom 50	Figure 4.5 : MCR during Measurement	40
Figure 4.7 : Tribometer During Experiment 42 Figure 4.8 : Density Meter during Experiment 43 Figure 4.9 : Electrode of pH Meter in Nano Drilling Fluid 43 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 46 Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 140°C (Bottom) 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) 49 Figure 5.4 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom) 50	Figure 4.6 : Heating Element in Test Fluid	41
Figure 4.8 : Density Meter during Experiment 43 Figure 4.9 : Electrode of pH Meter in Nano Drilling Fluid 43 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom)46 45 Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 140°C 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)49 47 Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C 48 (Bottom) 50	Figure 4.7 : Tribometer During Experiment	42
Figure 4.9 : Electrode of pH Meter in Nano Drilling Fluid 43 Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom) 46 Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 140°C (Bottom) 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) 49 Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom) 50	Figure 4.8 : Density Meter during Experiment	43
Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom)46 Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 140°C (Bottom) 47 Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)49 Figure 5.4 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)	Figure 4.9 : Electrode of pH Meter in Nano Drilling Fluid	43
Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 140°C (Bottom)	Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom).	46
(Bottom)	Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 140°C	
Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)49 Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)	(Bottom)	47
(Bottom)	Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom) Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C	49
	(Bottom)	50

Figure 5.5 : Effect of 0.6 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom	n)52
Figure 5.6 : Effect of 0.6 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C	
(Bottom)	53
Figure 5.7 : Effect of Graphene Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)	55
Figure 5.8 : Effect of Graphene Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)	56
Figure 5.9 : Effect of CNT Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)	57
Figure 5.10 : Effect of CNT Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)	58
Figure 5.11 : Effect of Nanosilica Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)	59
Figure 5.12 : Effect of Nanosilica Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)	60
Figure 5.13 : Effect of Alumina Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)	61
Figure 5.14 : Effect of Alumina Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)	62
Figure 5.15 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 2 at 30°C (Top) and 60°C	
(Bottom)	64
Figure 5.16 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 2 at 100°C (Top) and 140°C	
(Bottom)	65
Figure 5.17 : Effect of 0.6 weight% Nanoparticles Addition in Base Fluid 2 at 30°C (Top) and 60°C	
(Bottom)	67
Figure 5.18 : Effect of 0.6 weight% Nanoparticles Addition in Base Fluid 2 at 100°C (Top) and 140°C	
(Bottom)	68
Figure 5.19 : Effect of Graphene Addition in Base Fluid 2 at 30°C (Top) and 60°C (Bottom)	70
Figure 5.20 : Effect of Graphene Addition in Base Fluid 2 at 100°C (Top) and 140°C (Bottom)	71
Figure 5.21 : Effect of CNT Addition in Base Fluid 2 at 30°C (Top) and 60°C (Bottom)	72
Figure 5.22 : Effect of CNT Addition in Base Fluid 2 at 100°C (Top) and 140°C (Bottom)	73
Figure 5.23 : Effect of Nanosilica Addition in Base Fluid 2 at 30°C (Top) and 60°C (Bottom)	74
Figure 5.24 : Effect of Nanosilica Addition in Base Fluid 2 at 100°C (Top) and 140°C (Bottom)	75
Figure 5.25 : Effect of Alumina Addition in Base Fluid 2 at 30°C (Top) and 60°C (Bottom)	76
Figure 5.26 : Effect of Alumina Addition in Base Fluid 2 at 100°C (Top) and 140°C (Bottom)	77
Figure 5.27 : Effect of Nanoparticles Addition in Pure Water	79
Figure 5.28 : Effect of Nanoparticles Addition in Base Fluid 1	80
Figure 5.29 : Effect of Nanoparticles Addition in Base Fluid 2	82
Figure 5.30 : Effect of Nanoparticles Addition on Density of Pure Water	83
Figure 5.31 : Effect of Nanoparticles Addition on Density of Base Fluid 1	84
Figure 5.32 : Effect of Nanoparticles Addition on Density of Base Fluid 2	85
Figure 5.33 : Effect of Nanoparticles Addition on pH of Pure Water	86
Figure 5.34 : Effect of Nanoparticles Addition on pH of Base Fluid 1	87
Figure 5.35 : Effect of Nanoparticles Addition on pH of Base Fluid 2	88
Figure 6.1 : Temperature Profile inside Drill Pipe	91
Figure 6.2 : Temperature Profile inside Annulus	92
Figure 6.3 : Viscosity Profile inside Drill Pipe	94
Figure 6.4 : Viscosity Profile inside Annulus	95
Figure 6.5 : Fricton Factor Profile	97
Figure 6.6 : Drag Force in the Well with BF2	99
Figure 6.7 : Torque in the well with BF2	100
Figure 6.8 : Drag Force in the Well with 0.6%CNT in BF2	101
Figure 6.9 : Torque in the Well with 0.6%CNT in BF2	102

List of Tables

Table 2.1 : Various Definitions of Nanoparticles and Nanomaterials [13]	7
Table 2.2 : Physical and Chemical Properties of Nanoparticles [15]	7
Table 2.3 : Effect of Nanoparticle Addition in Drilling Fluid	11
Table 2.4 : Common Friction Factors [56]	26
Table 4.1 : Base Fluid 2 Composition	35
Table 4.2 : Nanoparticles and their Properties	37
Table 4.3 : Concentration Matrix of Nanoparticles	38
Table 5.1 : Mean Friction Factor with Pure Water	78
Table 5.2 : Mean Friction Factor with Base Fluid 1	80
Table 5.3 : Mean Friction Factor with Base Fluid 2	81
Table 6.1 : Viscosity Models for different Fluids	93
Table 6.2 : Frcition Factor Models for different Fluids	96

Nomenclature

F	Force (N)
Α	Area (m ²)
μ	Viscosity (Pa.s)
V	Velocity (m/s)
L	Length (m)
τ	Shear Stress (Pa)
γ	Shear Rate (s^{-1})
$ au_y$	Yield Stress (Pa)
μ_{pl}	Plastic Viscosity (Pa.s)
Θ_{600}	Dial Reading at 600 RPM
Θ_{300}	Dial Reading at 300 RPM
Κ	Consistency Index
n	Flow Behavior Index
$ au_o$	Yield Stress-Herschel Bulkley (Pa)
F _F	Force of Friction (N)
F _N	Normal Force (N)
Т	Time Period (s)
m_{C}	Empty Tube Mass (kg)
V_{C}	U-Tube Internal Volume
ρ	Density (kg/m^3)
$ ho_a$	Air Density (kg/m ³)
T_a	Time Period with Air (s)

Abbreviations

WBF	Water Based Fluid
HPHT	High Pressure High Temperature
LPLT	Low Presuure Low Temperature
DIF	Drill In Fluid
OBF	Oil Based Fluid
SBF	Synthtic Based Fluid
ECD	Equivalent Circulating Density
NNI	National Nanotechnology Institute
ISO	International Standards Organisation
NIOSH	National Institute for Occupational Safety and Health
BSI	British Standards Institute
ASTM	American Society for Testing and Materials
BAUA	Bundesanstalt für Arbeitsschutz und Arbeitsmedizin
NCT	Nano Computerized Tomography
CNT	Carbon Nano Tube
MCR	Modular Compact Rheometer

1 Introduction

Nanotechnology has emerged as tool and technology that has been implemented successfully in different fields including medicines, electronics equipments and composite materials. Nanoparticles are engineered in such a way that magnetic, optical and mechanical properties become enhanced so that they can be used in different applications. Various companies have also invested both money and time in order to get the benefits of nanotechnology in the field of drilling fluids. They have now realized several advantages of nano drilling fluids in the areas of rheology, wellbore stability, torque and drag, inhibition of shale and high temperature applications.

Highly deviated and Extended Reach Wells (ERWs) are among the recent advancements in drilling technology which have brought enormous number of benefits including greater reservoir exposure, extraction from thin layers, efficient stimulation and connecting the vertical fractures. But ERWs also offer various challenges during directional drilling and need sound planning. High torque and drag is one the primary challenge that can be experienced in ERWs. Torque and drag depends upon several factors such as mud density, hole inclination and azimuth and others. One of the most influential parameter of torque and drag is the frictional resistance between drill string and casing/open hole. Thus the problem of high torque and drag can be controlled by reducing friction factor in the well and thereby allowing ERW's to reach its maximum limits.

Nanoparticles have been used in several fields to provide better lubrication between the contacting surfaces. Different explanations have been given to support the friction reduction caused by nano fluid. Ball bearing theory is among them which states that nanoparticles acts rounded particles between two surfaces and prevent them to come in contact with each other [1]. In this way, it helps the contacting surfaces to slide over each other with relatively less frictional resistance. Another explanation of friction reduction by nanoparticles is through the development of smear film in between contacting surfaces [1].

The thesis aims to study the effect of various types of nanoparticles in water based drilling fluid on its tribological as well as rheological properties. Six different types of nanoparticles are used in the project which includes graphene, carbon nano tubes, silica, alumina, cobalt and nickel. Among them only alumina particle has particle size in micro scale. Each nanoparticle along with its properties such as size, surface area, density, purity is selected based on thorough literature survey done during the project. Two different concentrations of nanoparticles are tested in base fluids to analyze the effect of concentration on its behavior.

In addition to this, three base fluids are prepared in which nanoparticles have been dispersed. Initially effect of nanoparticles is determined in pure water as base fluid and results for rheology and tribology are generated through rheometer and tribometer respectively. Afterwards, a new water based drilling fluid is designed and prepared with small amount of salts and polymers. Then nanoparticles are mixed at particular concentrations to observe their influence on friction factor and viscosity of water base drilling fluid. A more complex and practical mud design is then prepared and a water based mud is made through this program which comprises of an alkali and weighting material in addition to salts and polymers. Similar procedures of nanoparticles addition and experiments are repeated with this base fluid. Results of viscosity and shear stress as function of shear rate are generated along with tribometer results which plot friction factor as function of temperature.

During experimental study, density and pH of nano drilling fluids are also evaluated in addition to rheology and tribology. Modular Compact Rheometer with high pressure cell apparatus is used in the project to evaluate the influence of nanoparticles on rheology at different temperature condition in a

confined environment. Friction factor offered by nano drilling fluid is measured with the help of tribometer based on pin-on-disc technology. The tribometer also enables determining friction factor at different temperatures.

Experimental study forms the foundation for temperature, viscosity, friction factor and torque and drag modeling work. Selection of nano drilling fluid is done on the basis of viscosity and friction factor results. All the models are implemented in computer software. Temperature Model is developed by using steady state heat transfer assumption which calculates temperature profile of drilling fluid inside drill pipe and annulus. Viscosity and friction factor models are generated by using experimental results at different temperature and regression analysis. These models utilize well bore temperature profile to determine viscosity and friction factor. Afterwards, friction factor profile is taken into three dimensional torque and drag model which produces torque and drag force profiles for static, hoisting and lowering cases.

The objective of the study is to reach conclusions on type and concentration of nanoparticles that has capability to reduce friction as well as torque and drag with sufficient viscous behavior so that hole cleaning do not get compromised. In addition, interaction of drilling fluid additive, such as polymers, with nanoparticles is also investigated in terms of change in rheological and tribological behavior of drilling fluid. Influence of density and pH of nano drilling fluid on its performance is also covered as part of study. The thesis also demonstrates the role of enhanced thermal conductivity of nano drilling fluid in cooling the drill bit.

2 Drilling Fluid and Nanoparticles

2.1 Introduction to Drilling Fluid

Drilling fluid, also known as mud, is one of the most important component of well construction process. It is designed to perform in the best way possible under predicted wellbore conditions. Nowadays, technology has made it possible to come up with highly efficient and cost effective drilling fluids that are suitable for different sections in the well.

Drilling fluid costs around 10% of the total tangible costs of well. But its performance can heavily affect the overall cost of well construction process. An efficient drilling mud can keep the cost under control by maximizing the rate of penetration (ROP), reducing possible loss of circulation, maintaining well bore stability, minimizing formation damage and keeping compliance with HSE requirements [2].

2.2 Basic Functions of Drilling Fluid

Drilling fluid serves many function during drilling as well as other operations such as tripping, logging and cementing. Few of them are highlighted in this section.

• Transport Cuttings to Surface

The basic function of drilling mud is to transport the cuttings from beneath the bit to the surface. The fluid is designed with such properties that help to carry the cuttings. Different solids are usually added to give those properties to drilling fluid. Correct Chemical properties are also necessary to avoid the dispersion of drilled solids. Otherwise, it can lead to generation of ultrafine particles which can affect drilling efficiency and productivity of pay zone.

• Prevent Well-Control Issues

The hydrostatic head of drilling fluid creates well bore pressure. This pressure should either balance or exceed the pore pressure of the formation to be drilled under normal conditions. As the drilling proceeds, pore pressure of the formation usually increases and sometimes it can go to abnormal pore pressures. The density of the drilling mud must be altered accordingly so that any possible well control situation can be avoided. Therefore selection of proper mud weights for each interval is vital to minimize kick.

• Preserve Wellbore Stability

Maintaining well bore stability is very critical for a successful drilling operation. Drilling fluid density and proper composition can result in a stable well bore which is very important for tripping drill string, running logging tools, conducting casing running and cementing operation.

• Minimize Formation Damage

Producing formations have continuous exposure to drilling fluid when they are being drilled. Drilling fluid has a tendency to lose its fluid filtrate and/or solids into the formation which in result alters the

productivity. A careful design of mud can minimize this formation damage and can be validated by testing it onto the core samples.

• Cool and Lubricate the Drill string

Circulation of drilling mud acts as a heat exchanger for bit and drill string and reduces the friction between tools and hole wall. The generation of heat is natural process dependent on the geothermal gradient of the area. Lubricity is required due to the directional trajectory of the wells as well as tight spots. Mud, utilizing oil as its base fluid, offers high lubricity and finds its application in high angle directional wells.

• Provide Information about the Wellbore

Drilling fluid is also used to provide the information about the wellbore. Mud pulse telemetry is the basic principle of measurement for transferring downhole survey and logging data during drilling. It also serves as a transferring medium during wireline logging operation. Preservation of cutting by drilling fluid and its efficient transfer to surface allow the geologists to analyze the cutting accurately [**2**].

2.3 Types of Drilling Fluids

Different types of fluid system exist including saltwater system, freshwater system, oil or synthetic based systems, and pneumatic (mist, foam, air, gas) system. Technical performance, environmental impact and cost are the major factors in selection of fluid system.

2.3.1 Water Based Fluids

The base fluid in this type of mud can be fresh water, seawater, saturated brine, simple brine, or formate brine. Almost 80% of wells are drilled by water-based fluids (WBFs). Well condition and specific interval of well being drilled determines the type of fluid. For instance, Sea water based fluid is typically used to drill surface intervals which contain fewer additives. Hole cleaning and fluid loss control are usually done by addition of commercial bentonite. In deeper section, same WBFs can be used depending upon well condition or can be replaced by oil or synthetic based system [**2**].

There are two major categories of water based fluid;

• Non-dispersed Systems

Example of non dispersed system are polymer systems with low/no bentonite and simple gel-water system that are use to drill surface intervals. Dilution, encapsulation and/or flocculation are used to manage natural clays in non-dispersed systems. Fine solids are removed from this type of system through a proper solid control system. In low solid non-dispersed polymer system, fluid rheology and fluid loss are managed by low and high molecular weight long chain polymer system. In HPHT applications, specially designed polymers are used so that they can remain stable at higher temperatures [2].

• Dispersed Systems

In dispersed systems, chemical dispersants are added to deflocculate clay particles so that rheology of high density muds can be improved. pH level is maintained at 10.0 to 11.0 by adding caustic soda

(NaOH). The mud weight of this type of system can be increased up to 20.0 ppg due to solid dispersion. Lignosulphonate system is an example of dispersed mud system [2].

There is another class in WBFs termed as salt water systems which are used to inhibit shale and to drill salt formation. Formation of ice-like hydrates can also be minimized by this fluid which can form around subsea equipment and well control equipment [2].

2.3.2 Drill-In Fluids (DIFs)

Conventional fluid can severely damage the reservoir productivity due to undefined risks associated with it particularly in horizontal wells due to long term exposure. Drill-in fluid is a type of fluid that is designed to mitigate formation damage and to offer better hole cleaning with easy cleanup. It can be based on water, oil, or synthetic systems. It also has a compatibility with reservoir fluid so that production of emulsions and precipitation of salts can be avoided. A detailed study of pay zones cores can be helpful in designing drilling fluid for reservoir [**2**].

2.3.3 Oil Based Fluids

These systems were developed in 1960s to minimize several drilling problems such as clay swelling, high bottom hole temperatures, high torque and drag, stuck pipe etc. The main constituent of OBF's is diesel, mineral oil or low-toxicity linear paraffin. The strength of emulsion is checked through monitoring of electrical stability of water phase. Oil based system utilizes barite as weighting agent and specially-treated organophilic bentonite as viscosifier. Other chemicals are used to control fluid loss, to suspend the particulate, to elevate pH, to mitigate the effects of H_2S and CO_2 gases. Typically 80/20 to 90/10 oil/water ratios is observed in field applications but in rare cases it can go to 95/5 as well [**2**].

One of the main benefits of oil based system is to inhibit shale from swelling. This is accomplished by high salinity water phase. Mostly calcium chloride is used to attain inhibitive property in oil based mud. The key issue with oil based system is their environmental impact. Offshore drilling does not allow the whole drilling fluid or cuttings to be discharged without processing. Therefore, there is always a cost associated with OBFs to process and ship waste fluid and cuttings [2].

2.3.4 Synthetic-Based Drilling Fluids

These fluid systems are developed to minimize the environmental effect that OBFs usually make by keeping the cost effectiveness of OBFs. SBFs offer shale inhibition, wellbore stability and better lubricity in directional wells. Therefore, drilling performance of both OBFs and SBFs are quite similar. The performance benefits include minimal initiation pressure to break the gel; very low equivalent circulating densities (ECDs); and nominal mud losses while drilling, running casing, and cementing. Regulations for cuttings generated through the use of SBFs are not as strict as it for OBFs [**2**].

2.3.5 All-Oil Fluids

High salinity water phase is normally used in invert-emulsion fluid to prevent swelling of reactive shales. Sometimes, long shale intervals are drilled with diesel or synthetic based oil with no water phase when there is a variation in formation water salinity. It helps to maintain shale stability in the whole interval.

2.3.6 Pneumatic-Drilling Fluids

Pneumatic fluids use air, gas, mist or foam to circulate cuttings out of the well. Specialized equipment are required when using this type of fluid to carry out operation in safe manner. These equipment aids in managing cutting and formation fluids on surface. Normally this type of fluid find its application in depleted or sub-normal pressure application and gives several benefits such as higher rate of penetrations (ROP) lost circulation prevention, hydrocarbon presence evaluation etc [2].

2.4 Nanotechnology

Nanotechnology is a recently developed field of science that is applied to study the matter on nanometer scale. Due to this, it is now possible to create new materials with better mechanical, optical and magnetic properties. This technology allows making structure unit of range from 1 nm to 100 nm. As the dimension of nanoparticle lies in the neighboring area between the clusters and the macroscopic materials, they will not directly demonstrate atomic and macroscopic properties, but bring with their own unique effects, for instance small size effect, surface effect, quanta size effect [3]. Therefore nanomaterials have specific characteristics in contrast to traditional materials, greatly enhancing the application areas of nanomaterials in various fields [4] [5] [6].

Nanotechnology can provide solution to the certain problem faced by drilling industry and also improve the overall performance of oil and gas sector [7] [8]. In the field of drilling fluid, nanotechnology can aid in maintaining bore hole stability and minimizing fluid loss which will lead to better and efficient drilling operation [9] [10]. The technology allows generating special characteristics in the drilling fluid which can work in complex environment to protect the reservoir from formation damage [11] [12]. Therefore, nanomaterials are nowadays considered as promising material to design and develop nano based drilling fluid which may offer optimized solutions to the problem that the conventional fluids cannot solve.

2.4.1 Definition of Nanoparticle

A particle of size between 1 and 100nm is termed as nanoparticle. Nanotechnology defines a particle as a small object that can act as a complete unit with regards to its properties and transport. Nanoparticle is a basic component in nano structural frame which is smaller than daily objects of world (Newton's law of motion) but larger in size than an atom or molecule (quantum mechanics). Figure 2.1 show how surface area can be improved by using nanoparticles instead of bulk material. After the development of National Nanotechnology Institute (NNI) in US in 2000, nanotechnology became the focal point for different media platform and got the attention from the community as well [13].



Figure 2.1 : Surface Area Modification using Nanoparticles [14]

As the size of particle is very small, both physical and chemical properties of these particles may differ from the bulk material. Surface areas, melting point, optical properties, magnetic properties, mechanical strength are among few of them. These properties also give an overview of why nanoparticles are used in industrial applications. Table 2.1 illustrates the definitions of nanomaterials and nanoparticles, stated by different organizations.

ORGANISATIONS	NANOPARTICLE	NANOMATERIAL
ISO	A particle spanning 1-100nm (diameter)	-
SCCP	At least one side is in the nanoscale range	Material for which at least one side or internal structure is in the nanoscale range
NIOSH	A particle with diameter between 1-100nm or fibre spanning a range of 1-100nm	-
BSI	All fields or diameter are in the nanoscale range	Material for which at least one side or internal structure is in the nanoscale range.
ASTM	An ultrafine particle whose length in 2 or 3 places is 1- 100nm	-
BAUA	All fields or diameter are in the nanoscale range	Material consisting of a nanostructure or nanosubstance

2.4.2 Properties of Nanoparticles

Nanoparticles, due to their small size, behave quite differently as compared to their origin material which is large in size. Table 2.2 presents a brief summary of major physical and chemical properties that a nanoparticle exhibit.

PHYSICAL PROPERTIES	CHEMICAL PROPERTIES	
Shape, size, aspect ratio, surface area	Molecular structure and structureal formula	
Aggregation/agglomertaion rate	Composition	
Particle size distribution and particle structure	Phase identity	
Topography/morphology of surface	Chemistry of surface (charge, reactie sites, zeta	
ropography/morphology of surface	potential, photocatalytic properties)	
Particle solubility	Lipophilicity/hydrophilicity	

2.5 Nanotechnology and Petroleum Industry

Various disciplines in petroleum industry are taking benefits from the development of nanotechnology. These phases include exploration, drilling, production, processing and refining. Subsurface imaging resolution has been made enhanced by incorporating nano sensors in exploration stage. Enhanced oil recovery method is also making the most of nanotechnology to get more hydrocarbons out of the reservoir

by modifying the molecules and by manipulating the interfacial properties. This section highlights the contribution of nanotechnology in various discipline of oil and gas industry to improve the performance.

2.5.1 Exploration

Use of nano sensors in exploration phase caught significant attention from petroleum geoscientists [16]. Imaging contrast agents and sensors can be developed with nanoparticles as they behave differently from their bulk counterparts with respect to magnetic, optical and electrical properties [6]. Deep wells exhibit hostile environments as the temperature and pressure are relatively high. A special type of sensor, called nano dust, is placed in the pore space to serve for several purposes such as recognition of fluid type, monitoring of fluid flow and characterization of reservoir [17]. Micro computerized tomography cannot detect effectively pore structure of tight formations. Thus nano computerized tomography (NCT) can be used to image these formations [14].

2.5.2 Drilling and Completion

Various fields of drilling phase are getting benefits from nanotechnology such as drilling fluid, drilling bit, down hole tools, cement and well logging. Use of nanoparticle in drilling fluid enables the engineers to maintain wellbore stability and control fluid loss specially in shale formation where the permeability is in nanodarcy (nD) [18]. Bit and stabilizer balling can be eliminated by using nano based drilling fluid as it creates a hydrophobic film on the surface [11]. Nano based drilling fluid can also aid in reducing torque and drag in extended reach and multilateral wells as they form an ultrafine thin film between pipe and hole wall [11]. This film provides ease when pipe is being rotated or tripped in/out of the well. Some nanoparticles in drilling fluid such as ZnO can be used to remove H_2S from water based mud before it reaches to surface [19]. It ensures reduction in pollution to environment, prevention of corrosion and protection of personnel.

In High pressure high temperature (HPHT) drilling operations, nano based drilling fluid system can perform efficiently as it contains particles of high surface area, large thermal conductivity, better interaction with rock and large heat tolerances [20]. These fluids can also reduce the wear and tear of down hole tools as they provide less abrasive force. From environmental perspective, nano based drilling fluid are also important as it utilizes very less concentration of nanoparticles which can be beneficial in sensitive environments [20].

Use of nano diamond in drill bit application and its integration into matrix of polycrystalline diamond compact (PDC) bit are studied by Chakraborty which can give unique characteristics to the bit [21]. As the engineered nanostructures have high mechanical strength, these structures can be used in making of down hole tools so that the tools can be run in hostile environments. Spacers with nano emulsions in cementing operations can also be optimized by using nanoparticles so that hole cleaning can be carried out effectively during cementing [22]. Several nanoparticles such as nanosilica, nanoalumina and Carbon Nano Tube (CNT) have been studied t improve the performance of cement with respect to its hydration process, fluid loss, gas migration and compressive strength [23].

2.5.3 Production

In production phase of an oil well, various application areas of nanotechnology are investigated by researchers such as hydrate recovery, scale inhibition and stimulation fluid. The recovery of hydrate can

be improved if the water cage decompose and release hydrocarbon (methane). This can be achieved by injecting Ni-Fe nanoparticles into hydrate formations if the particles are suspended in air having a self heating property [24]. In the field of stimulation, it is very common to use polymer based fluid containing high molecular weight cross linked polymer. As they produce large amount of residue, researchers are also studying the effect of low molecular weight surfactants as fracturing fluid with nanoparticle [25]. The nanoparticle gives desired properties to the fracturing fluid which helps in conducting efficient stimulation operation. Scale deposition in production tubing can be reduced by nano structures as they develop hydrophobic surface inside the tubing [26].

2.5.4 Reservoir Characterization

Enhanced oil recovery (EOR) has been focused from quite a while as the resources are moving towards the depletion phases. Nanotechnology has also brought its benefits to the field of EOR. As the size of nanoparticle is very small as compared to pore throat, they cannot be retained by the formation (at least after post flush). Aqueous dispersion containing nanoparticles displaces the discontinuous phase (oil, gas) due to the confinement of force of extremely large amount of nanoparticles at the vertex [27]. Oxides of zinc, aluminum, iron, magnesium, nickel, zirconium, silicon and tin are studied by Ogolo with regard to their application in EOR in which the particle size is kept to nano scale [28]. Fluid saturations can be evaluated by delivering paramagnetic nanoparticles carrying hydrophobic compounds can be injected into the reservoir and leave its hydrophobic component on its way to the recovery well if there is oil present in the reservoir [30]. It can enable us to determine the saturation of oil in the reservoir.

2.5.5 Refinery and Processing

Nanotechnology has been applied in the refinery for a long time especially with catalyst of nanometer size. Nanoparticles have a tendency to extract harmful substances like sulfur dioxides, nitrogen oxides and acids from the vapor. Nano membranes can be used to separate gas streams and to take impurities out from the oil [**31**]. Upgrading of heavy oil and bitumen can be done on site by using nano-catalyst so that the need to transport and handle them can be avoided [**32**].

2.6 Nano Drilling Fluid Research – A Brief Summary

Drilling fluid plays the similar role in the drilling operation as blood does in our body. Any issue in drilling fluid can severely affect the performance of whole operation. Therefore, it is always urged to understand the behavior and function of drilling fluid in the well. Drilling fluids are always being a focal point of many researchers so that improvement can be made in mud design and properties. Researchers are looking into the use of nano particles in drilling fluid because it can bring several benefits to mud. This section highlights the important work and milestone achieved in the field of nano-based drilling fluid so that the merits of nanotechnology can also be seen in this area of petroleum industry.

Contreras et al. discussed the use of iron based (Iron Hydroxide) and calcium based (Calcium Carbonate) nano particles in oil based mud (OBM) along with graphite as loss circulation material (LCM). They measured the rheological properties of drilling fluid both with and without nano particles and LCM at different concentrations. In the study, they found that calcium based nano particle gave high gel strength and plastic viscosity (PV) as compared to sample without nano particle. Iron based led to reduction in yield point (YP) at high graphite concentration but they did not affect plastic viscosity and gel strength significantly. Both of nano particles resulted in filtrate loss significantly and formed relatively thick mud cake [**33**].

Price et al. studied the use of graphene oxide (GO) and carbon nano tubes (CNT) as nano particles in drilling fluid. Graphene oxide was used in water based mud at different concentrations. Addition of GO after heat aging WBM at 150°C for 16 hours resulted in reduction in fluid loss at high concentrations but long term stability was an issue with GO. CNT was used in Synthetic Base Fluid at HPHT conditions (600°F). Significant low shear viscosity was observed along with better yield point & gel strength but fluid loss was still high [**34**].

Oscar et. al. studied the effect on wellbore strengthening by using nanoparticle with graphite in oil based mud (OBM). They observed an increase in fracture pressure by quantifying it on sandstone core using hydraulic fracturing tests. On using calcium based nanoparticles, fracture resistance was improved by 65% where as iron based nanoparticles increase it by 39%. Mud filtration was also studied in ceramic plate under HPHT conditions. The paper found out that the tip isolation mechanism associated with the production of immobile mass was responsible for higher fracture resistance [**35**].

Zakaria and his colleagues worked on the use of nanoparticles as loss circulation material (LCM). According to them, limited success is usually shown by micro and macro sized materials used in LCM particularly when the pore throat size is in micro scale. In-house nanoparticles were prepared either in OBM or in water. Low temperature and low pressure filtration tests were conducted with both fluid containing conventional LCMs as well as nano sized LCMs. It was found out that fluid loss got reduced by 70% through nano sized LCMs. Filter cake thickness was also measured through API standard procedure and better results were obtained by using nano-based drilling fluid [36].

Nanoparticles were also added to drilling fluid by Price et. al. to study the effect on shale permeability of two different shale formations. The idea was to understand the phenomenon of how pores got physically plugged by nanoparticles. Nanosilica particles of different sizes were selected based on the pore throat size of shale. The study also revealed that if the surface of nanosilica was treated then its performance in plugging the shale pores can be improved [**37**].

Fakoya et al. carried out experimental work to see the effect of nanoparticles on rheological properties of polymeric and surfactant based fluids. At different temperature, viscosity and frequency sweep test were conducted in rheometer by using 20 nm silica particles with its different concentrations. Maximum limit for concentration of nanoparticle which can improve the rheology was determined for both surfactant and polymer based fluids [**38**].

Table 2.3 also through some light on the effects of nanoparticle addition in drilling fluid which are experienced by different researchers in their experimental study.

NANO PARTICLES	CONCENTRATION (%)	TYPE OF DRILLING FLUID	EFFECT OF NANOPARTICLE ADDITION
Graphene Oxide	0.57-1.71%	WBM	Improve rheology, reduce fluid loss after heat aging, act as transport vehicle for placing stabilizers in shale, stability problem [34]
Carbon Nano Tube	0.14%	OBM	HPHT applications, Improve rheology, fluid loss problem, works very good with low concentrations [34].
NanoSilica	1-3%	WBM	Improve rheology, reduce filtrate loss, thin filter cake, works very good with high concentration [39].
Iron hydroxide	0.5-2.5%	OBM	Do not affect rheology significantly, considerable filtrate reduction under LPLT, better result with high graphite level, acceptable filter cake, lower concentration are efficient at HPHT [33].
Calcium carbonate	0.5-2.5%	OBM	Do not affect rheology significantly, considerable filtrate reduction under LPLT, better result with low graphite level, acceptable filter cake, higher concentration are efficient at HPHT but low at LPLT [33]

Table 2.3 : Effect of Nanoparticle Addition in Drilling Fluid

2.7 Comparison between Conventional and Nano Drilling Fluid

By using nanoparticle in drilling fluid, we can combat different drilling challenges. Following gives the highlights about it.

• Reduction in Formation Damage

Formation damage refers to alteration of formation characteristic typically due to drilling mud invasion. Due to this, pore volume and effective permeability tend to decrease near wellbore region. Formation damage is mostly due to spurt losses. Use of nano based fluid minimizes the spurt losses. It also protect the porosity/permeability characteristics of the near- wellbore reservoir section and increases productivity.

• Increase in Shale Stability

Shale formations are very complicated when it comes to solving the wellbore problems associated with them. Several chemical and mechanical actions are responsible for instability of reactive shale. Interaction between shale and mud can be minimized by using nanofluid because of its ultra fine particle size. It can also enhance the shale's resistance to fracture and collapse. Chemical reaction associated with shale-mud interaction can also be controlled using nanoparticles as it has several numbers of functional groups.

• Strengthening of Unconsolidated Formation

Unconsolidated formations are often encountered such as formations below deep sea bed due to low pressure from overlying rocks. Borehole problems are directly connected to the degree of unconsolidation. Conventional particles present in drilling mud are unable to generate effective inter particle cohesion and cementation as it comprises of macro or micro sized solid components. Nano particles can access to the pores and then to inter granular contact surfaces of unconsolidated sands. Use of nano particle increases fracture pressure thus strengthens the well bore.

• Formation of Ultrafine Mud Cake

Mud cake is usually formed on the face of permeable formation when a fluid is forced against the formation. It is very vital to know filtration properties and filter cake characteristics so that the downhole problems such as stuck pipe can be avoided. Conventional drilling mud comprises of micro and macro size particle which has a tendency to form thick filter cake. Nano-based drilling fluids form well dispersed, thin and tight mud cake, reduce the differential sticking problem, minimize torque & drag and decrease the scope of embedded cuttings bed formation in deviated, horizontal, and extended reach wells.

• Efficient in HPHT environments

HPHT environments have temperatures and pressures above 150°C and 690 bar respectively. Conventional macro and micro based fluids (chemicals and polymers) have limited thermal stability. They can get thermal degradation above 125-130°C. Due to degradation, these chemicals cannot perform their desired function effectively in the mud system. Therefore, in order to get desired viscous and gelling properties at high pressure and high temperature, drilling mud must comprises of the components that have stability under extreme conditions such as nanoparticles. Excellent thermal conductivity of nano based fluids with temperature and pressure tolerances can be a better choice

• Shallow Water Flow

Shallow water flow is potentially a problem in different wells especially deep water. It requires an additional casing to isolate them from other formation. It can also trigger several other borehole problems like instability of subsurface tools, fracturing of formation, well collapse, mud loss, erosion of seabed, mud properties alteration during well site operations [40]. Thus the cost of the well can be increased several folds due to well control issues, instability of rig, additional casing string and lost wellhead. As the size of nanoparticle is very small, it can penetrate the shallow water sands and result in improving bond strength between grains as it exhibits better cementing properties.

• Loss Circulation

Oil and gas wells frequently experience loss circulation problem due to several reasons. These reasons include unconsolidated nature of formation, presence of fracture network, and selection of improper mud weight especially when the window between pore pressure and fracture pressure is narrow. There are several loss circulation materials available in macro and micro sizes but there performance is not up to the mark. Customized nanoparticles can be made to act as structural barrier along the loss path so that effective sealing can prevent fluid loss. This will result in saving huge amount of revenue especially in case of oil based mud systems.

• Torque and Drag

In order to extract more hydrocarbons, the industry nowadays has inclined towards drilling horizontal and extended reach wells. But these well display enormous torque and drag during due to high frictional resistance between down hole tool and borehole wall. Therefore, drilling fluid which can give better lubrication between tools and wall can provide solution to the problem. Oil based mud lowers the frictional resistance but it is an expensive solution with environmental concerns as well. Other water based mud containing macro and micro sized particles can reduce the friction to very limited extent. The friction at pipe and wall interface can be significantly reduce by using nano based drilling mud as it form a thin and fine lubricating layer. A ball bearing effect, created by spherical nanoparticles also aid in sliding pipe in and out of the hole during tripping operation.

• Stuck Pipe

Both mechanical and differential pipe sticking can lead to an enormous increase in well cost. The risk of differential sticking can be easily reduced if drilling mud leaves a thin low sticking mud cake on the wall of borehole. Nano drilling fluid not only forms an ultrafine mud cake on the face of formation but also develop a non-sticking film on the down hole tools which helps to avoid triggering of differential sticking. In situation of stuck pipe, nanoparticles can be added to spotting fluid which will go inside the mud cake-pipe interface and release the stuck pipe. Conventional spotting fluid, comprising of bigger size than nanoparticle, find itself difficult to enter the interface.

• Bit Balling

Bit balling is quite frequent drilling problem especially in reactive formations such as gumbo shale. It not only creates problems for the bit but also for the stabilizers and tool joints. Due to bit balling, rate of penetration (ROP) gets drastically reduced as the clay accumulates in the tooth gaps of bit. Therefore, cost of drilling operation becomes high due to slower progress in drilling. Use of nano based drilling fluid can form a hydrophobic film which acts as barrier to bit balling.

• CO₂ and H₂S Environments

Acid gases such as CO_2 and H_2S can cause severe problems related to environment, process, tools, health and safety. H_2S gas in particular is deadly gas even at low concentration therefore, it is very important to control and treat it as quickly as possible during any operation. In drilling, formation may contain these gases and the solution is to neutralize them by using certain scavengers. Nano-based drilling fluid can be custom made to contain functional groups that can neutralize H_2S into less hazardous compound.

• Economics

Low cost is one of the most promising features of nano drilling fluid along with the other technical benefits. Small concentration of nanoparticle can yield extraordinary results in terms of performance. This is due to their high surface area which makes them highly reactive.

2.8 Potential Nanoparticles

This section covers brief summary of each nanoparticle that was used during the study.

• Graphene

Graphene is composed of thin layer of carbon atoms that are connected with each other in hexagonal structure (Figure 2.2). It is also an allotropic form of carbon which forms graphite if their layers stack on top of each other. Very small thickness, very light in weight, highly strong, best conductor of electricity and heat are among the major attributes of graphene. Since carbon is the 4th abundant element in the universe, therefore, graphene can provide ecologically friendly solutions to various applications such as composite materials, bioengineering, electronics, batteries, drilling fluids.



Figure 2.2 : Graphene [41]

• Nanosilica

Silicon dioxide or silica consists of two atoms of oxygen with a silicon atom having a molecular formula of SiO_2 . The most common form of silica is quartz. Nanoparticles of silica are highly stable, less toxic and can work effectively in the presence of other molecules. They are divided into p-type and s-type particle on the basis of their structure. P-type (porous type) has a nano pore rate of 0.61ml/g with several nano pores. Its surface area is relatively higher than s-type (spherical type). Ultraviolet reflectivity of p-type particle is also greater than s-type. The area of application of nanosilica is very vast. Some of them include plastics, paints, rubber, batteries, adhesives, concrete, fiber etc [42]. Figure 2.3 shows image of nano silica obtained from scanning electron microscopy (SEM).



Figure 2.3 : Image of Nanosilica obtained from Scanning Electron Microscopy (SEM) [43]

• Carbon Nano Tubes

Carbon nano tube (CNT) is one of the allotropic forms of carbon with cylindrical structure. It is composed of carbon atoms which are connected in hexagonal shapes. Every carbon atom is covalently bonded to its three neighboring carbon atoms. Length of CNT can be up to 132,000,000 times greater as compared to its diameter which is very high as compared to other materials. Due to cylindrical structure, certain properties of CNT are exceptionally improved such as mechanical strength, unique electrical characteristics and thermal conductivity. CNT belongs to fullerene family which also contains bucky balls. CNTs are classified into two types, designated as single-walled carbon nanotubes (SWCNT) and multi-walled carbon nanotubes (MWCNTs). SWCNT consist of only one cylinder of carbon atom (Figure 2.4) where as multiple concentric cylinders are present in MWCNT (Figure 2.5). CNT is one of the strongest materials in terms of elastic modulus and tensile strength. CNT typically shows hydrophobic property. Heat transfer capability of CNT depends upon the direction. Along the tube length, its thermal conductivity is very high but laterally to the axis of tube, it shows a good insulating property [**41**].



Figure 2.4 : SWCNT [44]



Figure 2.5 : MWCNT [44]

• Alumina

Alumina or aluminum oxide has molecular formula of Al_2O_3 which occurs naturally in crystalline form. It exhibits strong hardness with high melting point. Although it is an electrical insulator, its thermal conductivity is high enough for ceramic material. Nano alumina is also highly stable with respect to its dimension and phase. It is widely used in different applications such as rubber, plastics, ceramics etc. It can improve thermal fatigue resistance, creep resistance, fracture toughness and wear resistance [42].



Figure 2.6 : Nanoalumina [45]

• Cobalt nanopowder

Cobalt nanopowder is grey to black in color with spherical particles. They are produced from highly pure cobalt metal through laser evaporation process. The melting point of cobalt nanoparticles is 1495°C. It shows magnetic properties which make it suitable for imaging and sensors applications. They are also used in plastics, coatings, nano fibers and textiles. They are harmful with the risk to cause allergic skin reactions. On inhaling, they can lead to asthma and breathing problems [**46**].



Figure 2.7 : Cobalt nanopowder [47]

• Nickel Nanopowder

Nickel exists in the form of ore naturally as well as can be found as free metal. Different corrosion resistant alloys are being made by using nickel along with other metals such as chromium, molybdenum, iron and tungsten etc. Nickel nanoparticles can either be present in the form of nano fluid or in highly pure form as powder. They are prepared by thermally decomposing nickel acetylacetonate in alkylamines. For its characterization, transmission electron microscopy (TEM), X-ray Diffraction (XRD) and magnetic measurements are used. Its application areas include fuel cells, plastics, coatings, nano fibers and catalyst.



Figure 2.8 : Nickel Nanopowder [47]

2.9 Drilling Fluid Rheology

Rheology is the study of flow of matter and its deformation. It serves as source of extended knowledge for numerous industries such as food, chemical and oil industry. It is studied not only for drilling fluid but also for cements, work over and completion fluids. Rheological properties define the characteristics of flow of drilling fluid under various flow conditions. The prediction of this flow depends upon the knowledge of behavior of drilling fluid in the circulating system at different positions. This fluid behavior also characterizes drilling fluid and can answer the following two important questions;

- When does the fluid movement commence?
- What are the properties of fluid movement after it commencement?

2.9.1 Newtonian vs. Non-Newtonian Fluids

Frictional drag which is exerted by fluid flow on a surface of a pipe or conduit is termed as shear stress. It is a function of frictional drag that lies between adjacent layers and their velocity difference. Shear rate is the velocity gradient between two adjacent layers. Fluid can be classified as *Newtonian* and *non-Newtonian Fluids*. Newtonian Fluid exhibits a constant ratio between shear stress and shear rate which is termed as *viscosity* of the fluid. Therefore, measurement of both shear rate and shear stress at single point is sufficient enough to predict the behavior of the fluid. Water, high gravity oils, gases are among the examples of Newtonian fluids.

In order to completely understand the concept of viscosity, consider two plates of area A which are apart from each other at a distance L (Figure 2.9). Fluid is placed in between the plates which are initially at rest. The upper plate is then moved in x-direction with a velocity V. when the steady motion is attained, a force F is needed to maintain the motion of upper plate at velocity of V. the force F is given by,

$$\frac{F}{A} = \mu \frac{V}{L}$$

Where

$$\mu = viscosity of fluid$$

$$\frac{F}{A} = Shear Stress$$

$$\frac{V}{L}$$
 = Shear rate or velocity gradient



Figure 2.9 : Plate Experiment [48]

The relationship between shear stress and shear rate is linear as long as *laminar flow* prevails in which fluids use to move in layers. Only smaller values of shear rate can maintain laminar flow conditions. As the shear rate increases, laminar flow converts into *turbulent flow* in which fluid particles shows chaotic motion resulting in the formation of eddies and vortices.

The behavior of drilling fluids is similar to *non-Newtonian fluid* which does not show constant viscosity at different shear rates. Thus, there is no direct proportionality between shear rate and shear stress. if the apparent viscosity becomes high as shear rate increases, non-Newtonian fluids are termed as *dilatants* (*shear thickening*) and if it becomes smaller then they are called *pseudoplastic (shear thinning)*. If the apparent viscosity becomes smaller with time, non-Newtonian Fluids are called *thixotropic* where as if it increases with time then those fluids are *rheopectic*. Cements and drilling mud generally exhibit *pseudoplastic* and *thixotropic* behavior with respect to shear rate and time respectively. Figure 2.10 summarizes different type of fluid behaviors.


Figure 2.10 : Fluid Types [49]

2.9.2 Rheological Models

Rheological models are used to define the behavior of fluid when it is subjected to flow at different conditions. It comprises of constitutive equation with the constants that are determined experimentally. Different types of rheometer are now used to carry out the experiments. By putting the values of shear rate on x-axis and shear stress on y-axis, flow curves are generated which are then compared with flow curves of different rheological models so that best model can be selected to describe the fluid flow. Figure 2.11 demonstrates different rheological models developed with the span of time.



Figure 2.11 : Rheological Models [50]

• Newtonian Model

Newtonian models works for Newtonian fluids which exhibits direct proportionality between shear stress and shear rate. It means that the fluid movement begins as soon as the shear stress is applied to the fluid. Mathematically, Newtonian model is expressed as, $\tau = \mu \gamma$

Where

 $\mu = viscosity of fluid$ $\tau = Shear Stress$ $\gamma = Shear rate$

Bingham Plastic Model

This model defines the behavior of non-Newtonian fluids having a yield stress or point (YP) which is defined as the minimum shear stress that needs to be achieved before the fluid begins to flow. After this shear stress, the fluid will follow the behavior of Newtonian fluid which implies that viscosity will be similar as the shear rate changes. This viscosity is termed as plastic viscosity (PV). This models works better for the fluids having dispersed solid such as drilling fluids. The state of mud can be accurately described through this model but calculation of pressure and viscosity through this model does not match with the field values. The equation of Bingham Plastic Model is given by,

$$\tau = \tau_y + \mu_{pl}\gamma$$

Where

$$\mu_{pl} = Plastic Viscosity (PV)$$

 $\tau_v = Yield Point (YP)$

The values of PV and YP are calculated by using two different RPM reading (600 and 300) on viscometer.

$$\mu_{pl} = \Theta_{600} - \Theta_{300}$$
$$\tau_y = \Theta_{300} - \mu_{pl}$$

• Power Law Model

This model works better for the drilling fluid containing polymers with the aim to eliminate the defects in Bingham plastic model at low shear rates. The relationship between shear stress and shear rate in nonlinear according to this model. Similar to Newtonian model, it also assumes that the fluid movement will commence as soon as the shear stress is applied which is a drawback of using this model for drilling fluids. Model uses two parameters k and n to describe the fluid behavior.

$$\tau = K\gamma^n$$

Where

K = Consistency Indexn = Flow Behaviour Index The value of flow behavior index decides the nature of fluid as Newtonian (n = 1), pseudoplastic (n < 1) or dilatant (n > 1). The value of n and K can be determined by using two RPM reading (600 and 300) on viscometer.

$$n = 1.4427 * \log \frac{\theta_{600}}{\theta_{300}})$$
$$K = 1.067 * \frac{\Theta_{600}}{1022^n}$$

• Herschel-Bulkley Model

It is also named as modified power law as it combines Bingham plastic model with power law model. It is also used to describe the behavior of pseudoplastic fluid. Unlike to power law, it considers yield stress in the model and thus can describe the measured value to shear stress and shear rate more accurately than others. Model is represented as,

$$\tau = \tau_o + K \gamma^n$$

Where

$$\tau_o =$$
 Shear stress at zero shear rate

2.10 Drilling Fluid Tribology

It is a science which defines how elements behave in the presence of friction. There is always friction present between the materials when they are in contact with each other. The magnitude of friction between elements depends upon several factors such as shape, asperity and abrasiveness. It is obvious to mention the relationship between friction and wear. Therefore, tribology not only includes studying the frictional forces but also wear and lubrication.

When solid surface interact tribologically with other materials, loss of material occurs which is known as wear. Adhesion, fatigue, erosion and abrasion can be called as major types of wear. Among them, abrasive and adhesive wear phenomenons are of great interest with respect to this study. The amount of wear depends upon surface properties and presence of lubrication between the interacting materials.

2.10.1 Wear Phenomenon

There are four major types of wear phenomenon.

Abrasive wear

In drilling, both impact and sliding movement can be experienced when down hole tools are in relative motion to the hole wall. It forms the basis of *abrasive wear*. This type of wear generates particles which are stronger than parent surface and can cause further wear in the system. Inside the clearance, particles work like cutting tools and remove the matter from the opposite surface Figure 2.12. Abrasive wear can eventually lead to changes in dimensions, lower efficiency and leaks. This type of wear is also experienced when conducting pin-on disk experiment during the study which is described in Section 5.2. There are some derived forms associated with abrasive wear such as polishing, grinding and machining.



Figure 2.12 : Abrasive Wear [51]

• Adhesive wear

Adhesive wear occurs when material interact with another material due to low speed, high loads and /or viscosity reduction. Asperities on surface are generally cold welded with each other and movement cause the particle to shear off. Figure 2.13 presents an example of adhesive wear between two surfaces.



Figure 2.13 : Adhesive Wear [51]

• Fatigue wear

Fatigue is another type of wear mechanism which is defined by the fact that repeated friction impact can create crack in the material which can eventually lead to its failure. The particles actually get trapped inside the clearance and cause repeated stress. At first cracks are formed on the surface which then spread in the whole system to fail it. Figure 2.14 depicts how fatigue wear happens.



Figure 2.14 : Fatigue Wear [51]

• Erosive wear

Erosive wear is associated with particles that forces themselves on the edges or surfaces and break the material from it due to their momentum (Figure 2.15). The system having high velocity flows generally exhibits erosive wear. This type of wear is very detrimental to the whole system as the particle can cause dimensional changes, reduction in system efficiency, leakages and production of new particles which will trigger wear in the efficient part of the system.



Figure 2.15 : Erosive Wear [51]

During drilling, components of drill string such as drill pipes, drill collars, stabilizers, heavy weight drill pipe and other down hole tools have a tendency to come in contact with hole wall a well as casing which can lead to wear. Due to this, drilling fluid has to perform another function, in addition to other, which is to provide adequate lubrication so that the wear of tools can be minimized. This lubrication also aid to reduce several other drilling problems such as high torque and drag in directional wells.

2.10.2 Friction

The concept of friction was first put forward by Aristotle which was later stated by Leonardo da Vinci as the friction is independent of the length and breadth of contact between moving objects. Based on the work of Amontons and Coulomb, three laws of friction have been described [52],

- 1. Frictional force is proportional to load applied on the surface
- 2. Frictional force is not a function of contact area between the surfaces.
- 3. Kinetic Friction does not depend upon the velocity.

The term kinetic friction is a type of friction which occurs when contacting objects are in relative motion. Other type of friction is termed as static friction in which there is no relative movement between the objects. Figure 2.16 shows the behavior of both types.



The co-efficient of friction μ depends upon force of friction F_F and normal force F_N and is given by

$$\mu = \frac{F_F}{F_N}$$

It represents the degree of roughness that is present between the surfaces such as outer surface of drill string and inner surface of casing. Friction factor during drilling depends upon numerous factors [54]. Few of them are listed below,

- Viscous Drag
- Presence of cutting beds
- Loss of lubricity due to loss circulation
- Stiffness of pipe
- Wellbore tortuosity
- Well geometry
- Hole cleaning

The accurate knowledge of friction factor is very vital for conducting safer drilling operation. It plays an important role in both vertical as well as in deviated wells. Figure 2.17 presents a scenario in which friction between drill string and extended reach wellbore can generate energy and wear in the system. In vertical wells, contact between tubular and borehole wall is negligible which can result in almost no torque and drag. When it comes to deviated sections, both torque and drag begins to develop in the system as the tubular are pressed against the wall (Figure 2.17). High values of torque and drag can pose many limitations on the drilling operations and affects the overall performance. In these scenarios, low friction factors between tubular and wall can provide a solution which can lift those limitations and wells can be drilled deeper and cheaper.



Figure 2.17 Extended Reach Well [55]

2.10.3 Factor affecting Tribological Behavior

The tribological behavior depends on fluid composition with the following critical parameters:

• Particles shape and size

Sharpness on the edges of particles results in high degree of interaction with asperity of material as compared to edges with rounded or spherical profiles. Bigger size particle in drilling mud or lubricating fluid can enhance abrasive wear on the surfaces.

• Particles Concentration

As the concentration of particles increases in drilling fluid, the chances of wear usually increases due to more contact time of particle with the surface.

• Particle Solidness

As the down hole tool exerts various types of forces on both casing and hole wall, particles of drilling fluid must show some solidness. If the particles in drilling fluid are added by carefully examining the shape such as spherical, it can help to reduce the wear.

• Asperity of surface

Abrasive surfaces have a tendency to show high degree of wear.

• Types of forces

Different types of forces show different behavior when it comes to wear. Forces like lateral force, torsional force and hydraulic pressure can cause different types of wear.

• Time frame of the material contact

As the time of contact between the surfaces increases, lubrication tends to go down and results in high wear.

• Base fluid

For drilling fluid, it is very important to know whether it is water or oil-based. Oil based muds are relatively better in performance when it comes to lubrication and wear reduction. Table 2.4 shows a comparison between cased and open hole friction factors using different types of drilling fluids.

TYPE OF DRILLING	FRICTION FACTOR		
FLUID	CASED HOLE	OPEN HOLE	
Oil Based Mud	0.16 - 0.20	0.17 - 0.25	
Water Based Mud	0.25 - 0.35	0.25 - 0.40	
Brine	0.30 - 0.40	0.30 - 0.40	
Polymer based Mud	0.15 - 0.22	0.20 - 0.30	
Synthetic Based Mud	0.12 - 0.18	0.15 - 0.25	
Foam	0.30 - 0.40	0.35 - 0.55	
Air	0.35 - 0.55	0.40 - 0.60	

Table 2.4 : Common Friction Factors [56]

3 Experimental Setup

In order to study rheology and tribology of the drilling fluid, an experimental setup is selected consisting of modular dynamic rheometer (MCR) for rheology, pin on disk Apparatus for tribology, density meter for density and pH meter for pH measurement. Each of the instruments has its own procedures specified by different standardization authorities. During the experiments, care is taken to follow those procedures so that results can be compared and reported. User manuals as well as professional experts also play an important role in conducting experiments. Both of the sources are taken into consideration while doing measurements.

3.1 Rheometer

Rheology of drilling fluid is studied with the help of viscometer. Viscometer enables us to determine rheological properties of the drilling fluid. It primarily consists of cylindrical cup and bob. The rotation given to the bob is transferred to fluid filled in the cup as the bob is completely immersed in the fluid. This rotation generates drag force in the drilling fluid. The state of fluid prior to motion can affect the drag force. Viscometer are thus used to determine the behavior of drilling fluid. Different types of rotational viscometer are available which allow shearing of sample drilling fluid for a long time interval so that transient effect can be monitored or a state of equilibrium can be attained. Rotational measurement can be done in two ways; shear stress controlled and shear rate controlled. In stress controlled technique, rotation is generated by applying constant torque to the tool and speed of rotation is determined which is further used to calculate shear rate. In rate controlled method, torque is determined by maintaining rotational speed in the sample.

Rotational measurement systems comprises of four components

- 1. Measuring tool whose geometry is clearly defined.
- 2. Device to generate torque or rotational speed corresponding to shear stress and shear rate.
- 3. Device to calculate the response of shear stress or shear rate.
- 4. Device to maintain and control temperature during the measurements

Most of the rheometers consist of cylinder, parallel plate or cone and plate geometry (Figure 3.1).



Figure 3.1 : Schematic Diagram (a) Concentric Cylinder, (b) Cone and Plate, (c) Parallel Plate [57]

Cone and plate comprises of inverted cone which is in contact with the lower plate. Usually cone is maintained at 4° or less. Rotation can be given either to lower or upper surface. The simplified form of cone and plate is geometry called parallel plate with an inclination of 0°. Fluid sample is placed in the gap of two surfaces. Concentrated suspensions, gels and paste with high viscosity are studied using both of these methods. The third and most important geometry is concentric cylinder which allows rotating the cylinder in the fluid which is present in the clearance between the cylinders. The system is described by ISO 3219 (1993) and DIN 53019-1 standards [**58**]. Different configurations can be maintained with this geometry (Figure 3.2) Double gap scheme is suitable for less viscous fluid as it allow having more viscous drag due to large area of contact with the fluid. Cone and hollow cavity geometry reduces the end effects from the measured data.



Figure 3.2 : Three Alternative Cylindrical Tool Designs (a) Double Gap, (b) Cone and Plate at the Bottom, (c) Hollow Cavity at the Bottom to Trap Air [57]

In the study, concentric cylinder geometry with double gap configuration is used to study the rheology of nano fluid so that the accuracy of the experiment is ensured. Figure 3.3 shows Anton Paar's MCR equipped with pressure cell which is selected as the rheological behavior has to be studied under sealed environment to eliminate the risk of vaporization at high temperature. MCR is one of the recent rheometers that are fitted with patented senor for normal force, friction bearing and electronically commutated motor. It also allows both oscillatory and rotational measurements to be done using the same rheometer.



Figure 3.3 : Modular Compact Rheometer

Various components are present in pressure cell assembly (Figure 3.4). A brief description of each component is as follows,

• Magnetic coupling

Pressure head receive torque of measuring device through magnetic coupling which is then conveyed to measuring cylinder

• Pressure head

It provide sealed environment in the cell and confined the applied pressure to the cell. Rotating components are present in pressure head.

• Measuring system

Measuring system consists of measuring cup and measuring cylinder. As mentioned earlier, double gap measuring system is used in the study which contains an additional inner cylinder so

that the cross section depicts annular clearance (Figure 3.5). According to DIN 54453 standards, following two conditions must be satisfied during measurement;

$$\circ \quad \delta_{cc} = \frac{R_4}{R_3} = \frac{R_2}{R_1} \le 1.15$$
$$\circ \quad L \ge 3R_3$$

• Pressure cup

Drilling fluid sample is held inside the pressure cup which has connections for safety valve and pressure supply.

• Pressure supply unit

It consists of two valves; one for applying inlet pressure to the cell and other is for depressurizing the cell. Pressure source is also connected to this unit



Figure 3.4 : Pressure Cell Assembly [59]



Figure 3.5 : Double Gap Measuring System [59]

3.2 Tribometer

Lubrication behavior of any type of fluid can be studied by using pin-on disc equipment. It is industry standard to measure wear and friction. In this study, it is used to measure the friction factor between the steel ball and the steel plate in the presence of nano-based drilling fluid. DIN 50324 and ASTM G99 are used as standard during the experiment. As the name of equipment implies, it consists of pin which can be either spherical shaped or cylindrical shaped. Figure 3.6 illustrates forces in tribometer during measurement of friction factor. A disc (open one sided cylinder) holds the fluid sample on top of it. The diameter in which pin has to rotate is selected before starting the test. The applied force on the disk can also be adjusted by using different configuration weights. The body of the elastic arm consists of strain gauges bonded to it. By using elastic arm deflection, friction factor at different temperatures with the help of heating element. Figure 3.7 shows tribometer from CMI Instruments used in the study.



Figure 3.6 : Forces in Tribometer [60]

Following are the important features associated with pin-on disc equipment

- It enables travelling for infinite number of times at different linear velocities which depends on the diameter in which pin rotates.
- It can accurately model the lubrication behavior for parts having linear relative velocity.
- The edge of the pin might burrow in the disk and can affect the conditions of sliding



Figure 3.7 : Tribometer

3.3 Density Meter

Density of drilling fluid is measured by using Anton Paar's density meter (Figure 3.8). The working principle of density meter is oscillating U-tube technique. Fluid is filled inside the U-tube which is placed to vibrate. These vibrations have Eigen frequency associated with it which depends upon the mass and therefore on the density of fluid filled in the tube. This principle can be understood by considering an example of ball and leaf spring system as shown in Figure 3.9. On pressing the spring down and releasing it, oscillation starts in the system whose frequency depends upon the ball weight. Lighter ball vibrates with higher frequency than heavier ball. Thus, high density ball will demonstrate low frequency of vibration.



Figure 3.8 : Density Meter

U-tube inside the density meter also works in the similar manner. If high density fluid is filled in it, the frequency of oscillation will be small. For example, water (high density fluid) vibrates with low frequency as compared to air (low density fluid). The frequency of vibration of U-tube is given in terms of time period as [61],

$$T = 2\pi \sqrt{\frac{m_c + \rho V_c}{K}}$$

Where,

$$m_{C} = Empty \ tube \ mass$$

 $V_{C} = U - tube \ internal \ volume$
 $ho = Density \ of \ fluid \ in \ cell$
 $K = Specific \ constant \ of \ U - tube$

If the calibration factor F is determined, density of fluid can be calculated as [61],



Figure 3.9 : Oscillation of Leaf Spring System

3.4 pH Meter

Mettler Toledo's pH meter is used to determine the pH of drilling fluid during the whole study (Figure 3.10). SevenCompact series provide accurate and reliable measurement of pH. It consists of an electrode whose sensor has to be immersed in the fluid during measurement. The electrode is kept vertically straight with the help of electrode arm. The instrument measures pH by measuring the voltage of drilling fluid in which the sensor of electrode is dipped.



Figure 3.10 : pH Meter

4 Experimental Procedure

This chapter discusses the method that has been adopted during the experimental work of the thesis. Different laboratories of University of Stavanger (UiS) are used to study the effect of nanoparticles on rheological and tribological behavior of drilling fluid. Rheological measurements are determined by Modular Compact Rheometer (MCR) 302 model, manufactured by Anton Paar which works on the ISO 3219 (1993) and DIN 53019-1 standards of concentric cylinder measurements. Tribology lab is used to study the effect of nanoparticle on friction factor of drilling fluid with the help of pin-on disc apparatus which follows DIN 50324 and ASTM G99 standards. Anton Paar's density meter Model DMA-4500 is employed during density measurement. The measurements are done on the basis of oscillating tube standard (ISO 15212-1). For pH measurement, highly reliable pH meter is used manufactured by Mettler Toledo.

4.1 Nano-Based Fluid Preparation

Three fluids are tested as base fluid during the study, designated as Pure Water (PW), Base Fluid 1 (BF1) and Base Fluid 2 (BF2). Both BF1 and BF2 are water based drilling muds in which nanoparticles are added according to pre-determined concentration. Water based mud is selected due to its low cost and environmental friendly nature. The study covers the addition of different types of nanoparticles in drilling fluid and its effect on rheological and tribological behavior at various concentrations. These nanoparticles include graphene, silica, multi-walled carbon nano tubes, alumina, cobalt and nickel.

4.1.1 Base fluids Preparation

Pure water (PW) is used as first base fluid and no other chemicals are added to it. BF1 is a very simplified water base fluid which is made up of 18 ppb KCl (Potassium Chloride), 0.35 ppb of Xanthan Gum and 0.70 ppb of PAC UL (Poly Anionic Cellulose Ultra Low). KCl is normally added to drilling mud in order to acquire shale inhibitive property. Function of adding Xanthan gum and PAC UL polymers is to give viscous and filtration control properties respectively to BF1. The amount of each chemical is measured using precision balance as shown in Figure 4.1 and then mixed through in water using mixer (Figure 4.2). Another base fluid (BF2) is also studied during the project which comprises more chemicals in order to control other properties of drilling mud. Table 4.1 summarizes the composition, concentration, and function of each chemical in BF2. Weighting and mixing procedure and equipment are similar for BF2 as well.

S.NO	COMPONENT DESCRIPTION	CONCENTRATION (PPB)	FUNCTION
1	Calcium Carbonate (CaCO ₃)	65	Weighting/Bridging Material
2	Potassium Chloride (KCl)	18	Shale Inhibitor
3	Xanthan Gum	1.5	Viscosifier
4	Poly Anionic Cellulose (PAC UL)	3	Filtration Control Additive
5	Sodium Carbonate (Na ₂ CO ₃)	0.5	Calcium Precipitation
6	Sodium Hydroxide (NaOH)	0.25	Source of Alkalinity

Table 4.1	:	Base	Fluid	2	Composition
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Figure 4.1 : Precision Balance



Figure 4.2 : Mixer

4.1.2 Nanoparticle Addition

During every experiment, base fluid is prepared prior to addition of nanoparticles to it. The nanoparticles selected for the study have not been used before in drilling fluid specifically to the base fluid used here. Table 4.2 illustrates the complete information about each nanoparticle and Table 4.3 defines the concentration of each nanoparticle in different base fluids.

S.NO.	NANOPARTICLES	MANUFACTURERS	PROPERTIES	IMAGES
1	Graphene	American Elements	Structure : Multilayer Thickness : 10-15nm Average Diameter : 15µm Purity : 99+%	
2	NanoSilica	XF NANO INC.	Particle Size Range : 20nm Specific Surface Area : ≥600m²/g Purity: ≥99%	
3	MWCNT	Shenzhen Nanotech Port Co., Ltd. (NTP)	Diameter Range : 10- 20nm Length : <2µm Specific surface Area : 100-160m ² /g Purity : >97%	
4	Alumina	Sasol Germany GmbH	Particle size Range : 25-125 µm Specific Surface Area : 206 m ² /g Loose Bulk Density : 0.75g/ml	
5	Cobalt Nanopowder	Alfa Aesar	Average Particle Size : 25-30nm Purity : 99.8%	
6	Nickel Nanopowder	Sigma-Aldrich	Average Particle Size : <100nm Purity : ≥99% Density : 8.9g/ml	

Table 4.2 : Nanoparticles and their Properties

S.NO.	NANOPARTICLE	CONCENTRATION OF NANOPARTICLES IN BASE FLUIDS (WEIGHT %)			
		PURE WATER (PW)	BASE FLUID 1 (BF1)	BASE FLUID 1 (BF2)	
1	Graphene	0.6	0.2 and 0.6	0.2 and 0.6	
2	NanoSilica				
3	MWCNT				
4	Alumina Nanopowder				
5	Cobalt Nanopowder		-	-	
6	Nickel Nanopowder		-	-	

Table 4.3 : Concentration Matrix of Nanoparticles

4.2 Viscosity Measurements

Modular Compact Rheometer (MCR), available in the instrument lab at Petroleum Engineering Department at UiS, enables studying rheological behavior of drilling fluid at different conditions of pressure and temperature using different measuring systems. The instrument is also equipped with relevant computer program to start, control and monitor the experiment. For measuring rheology at different temperature, double gap measuring system (DGMS) is being used which employs pressure cell component during measurement (Figure 4.3). This system reduces the chances of vaporization of drilling fluid during the experiment due to high temperatures. Thus the concentration of additives in the fluid remains constant.



Figure 4.3 : Pressure Cell Components and other Accessories

In order to begin the test, all the fluid interacting components of pressure cell are cleaned with water and dried so that the risk of contamination from previously used fluid. The pressure cell is assembled by

placing the inner part of DGMS inside pressure cup and tightening it with the help of nut and special wrench. Specific volume of fluid (5.8 ml as per instruction manual) is filled in the cup by using filling adapter and measuring cylinder is then inserted into the cup after mounting it on pressure head. The connection at upper end of the pressure head is then tightened with the help of special wrench. Figure 4.4 depicts the pressure cell at the end of components assembling and fluid filling



Figure 4.4 : Pressure Cell with Assembled Components

After assembling the pressure cell, it is placed in the rheometer and all the necessary connections are made. These connections include temperature sensor (Pt100 Cable), pressure tube and silicone hose. All the measurements are done at slightly higher pressure so that dehydration of fluid can be minimized. The magnetic coupling is then attached to the rheometer and lowered using computer program so that the rotation can be transferred to the measuring cylinder. Relevant program for DGMS is selected in the software and a measurement scheme is designed in it. The scheme includes measuring shear stress and viscosity at four different temperatures (30 °C, 60 °C, 100 °C and 140 °C) under a given range of shear rate. The scheme gives enough time during experiment which ensures that measurement is performed after the fluid temperature has been reached to its planned value. Figure 4.5 illustrates an image of MCR during measurement.



Figure 4.5 : MCR during Measurement

4.3 Friction Factor Measurement

In order to study tribological behavior of nano drilling fluids, pin-on disc tribometer from CSM Instrument is used in Tribology Lab at UiS. The equipment along with computer program allows measuring friction factor between ball and steel plate in the presence of drilling fluid at different conditions of temperature. The test begins with removal of previously used drilling fluid from the one-sided open cylinder which is further followed by cleaning and drying of pin, disc and inner surfaces of cylinder. For every measurement, condition of steel ball inside the pin and steel disc is inspected and necessary alterations/replacements are made so that the measurements remain reliable such utilization of unused surface of ball and diameter of pin rotation on disc. Heating element is also positioned in the

cylinder to vary temperature during the experiment and cable is connected to the instrument so that temperature gets recorded during the test.

Prior to filling fluid, it is always made sure that the pin holder's arm remains perfectly horizontal during the experiment. A sufficient volume of nano drilling fluid is prepared according to the procedure described in Section 4.1 and cylinder is filled with it such that fluid completely covers disc as well as heating element. Figure 4.6 shows an image of heat element immersed in the sample fluid.



Figure 4.6 : Heating Element in Test Fluid

A measurement scheme is designed in the computer software for studying tribological behavior in which values for parameters are selected. These parameters include sample temperature, load on top of pin, distance travelled by pin and linear speed. For each fluid sample, friction factor is measured at four different temperatures (30 °C, 40 °C, 50 °C and 60 °C) under a load of 10 N with linear speed of 2.92 cm/s. Room humidity and temperature are also entered prior to starting any test.

The fluid inside the tribometer sample holder is then heated up to the sample temperature which can take several minutes. During the heating process, the sample holder along with fluid and disc is constantly rotating so that the fluid temperature remains uniform as well as rotation keeps fluid content well dispersed. After attaining required temperature, the experiment begins by lowering down pin arm into fluid and load is placed on top of the pin (Figure 4.7). During the test, it is always made sure that the factors leading to poor measurement or inaccurate results such as noise, stalled equipment are carefully monitored. The test is repeated if the measurement of friction factor gets interrupted by any factor. This procedure is repeated for each fluid, whether base or nano and friction factor curves are obtained at different temperatures.



Figure 4.7 : Tribometer During Experiment

4.4 Density Measurement

Instrument lab in Petroleum Engineering Department at UiS also has facility to measure density of nano drilling fluid using density meter, manufactured by Anton Paar. The procedure starts by setting up the software and temperature is maintained at 20°C. The instrument has a U-tube for measurement which has to be cleaned by acetone before commencing the test. Small volume of nano drilling fluid is injected into the instrument and the value of density is read on the screen after few minutes. Figure 4.8 demonstrates how fluid injection is done.



Figure 4.8 : Density Meter during Experiment

4.5 pH Measurement

The alkalinity of drilling fluid is an important property that can affect the performance of drilling fluid. In the study, pH measurements are incorporated by using Mettler Toledo's pH meter available in Instrument Lab at UiS. Prior to using pH meter, it is always ensured that the instrument is calibrated in recent times. The value of pH for different nano drilling fluid is obtained by immersing the sensor of the electrode in it for few minutes (Figure 4.9).



Figure 4.9 : Electrode of pH Meter in Nano Drilling Fluid

5 Results and Discussions

This chapter comprises of the results obtained from the experimental study. Rheology and tribology of three base fluids, designated as pure water (PW), base fluid 1 (BF1) and base fluid (BF2), are studied with different type and concentration of nanoparticles. Each fluid first prepared in the mixing laboratory and is then tested in the MCR and Tribometer at the same time. Density and pH of the drilling fluid sample are also measured. A relatively large amount of data sets are generated from the experiments which are attached in the appendices.

This chapter also presents the explanation behind the results obtained from all the experimental work. It also covers the relationship between the measured properties and its significance in drawing conclusions from the tests. The data from viscosity, fiction factor, density and pH measurements is analyzed on the basis of scientific phenomenon and the final analysis is presented in this chapter.

5.1 MCR Measurements

The effect of nanoparticle on rheological behavior of drilling fluid is studied using three reference fluids at different temperatures (30°C, 60°C, 100°C and 140°C). This section presents the results of shear stress and viscosity obtained through those fluids.

5.1.1 Pure Water

Six different types of nanoparticles are tested in pure water at concentration of 0.6% by weight. The temperature is setup in such a way that the rheological behavior of test fluid is recorded at four temperatures. Figure 5.1 and Figure 5.2 show the results of shear stress and viscosity with varying shear rate at different temperature with nanoparticles in water. In all figures, the viscosity decreases with temperature and also with increase in shear rate.

At 30°C (Figure 5.1), graphene addition to water result in high viscosity at all shear rates. The higher viscosity response is more prominent between 20 and 200 s⁻¹ shear rates. CNT, nanosilica and nickel shows similar amount of shear stress as with water. The behavior of cobalt and alumina is different. Shear stress of these nano fluids is initially higher than water till 200 s⁻¹. Afterwards, both fluids follow the trend of water.

Figure 5.1 also presents the effect of nanoparticles addition in pure water at 60°C. Nano fluid containing 0.6% graphene also shows higher shear stress and viscosity at 60°C as compared to water. CNT, nanosilica and nickel performs in similar manner as they do at 30°C. Alumina and cobalt nano fluids also give almost identical response as with pure water except at shear rates between 10 to 20 s⁻¹.

Shear stress and viscosity of nano fluid containing graphene is higher than that of water at 100°C (Figure 5.2). At high level of shearing (200-1020s⁻¹), all the other particles demonstrate almost similar level of viscosity as with only pure water. At lower shear rates, the response of all the particles fluctuates around water values.

The effect of graphene addition continues its similar trend at 140°C as well (Figure 5.2). Pure water containing CNT also shows higher level of shear stress and viscosity than water especially at lower shear rates where the values are even higher than pure water containing graphene. Nanosilica addition also follows the trend of graphene addition. Alumina, cobalt and nickel show similar level of viscous behavior as that of water.

In general, the addition of nanoparticle to pure water introduces non-Newtonian behavior in water. It can be observed that graphene addition at concentration of 0.6% by weight in water results in improving its rheological behavior at all temperatures because of the presence of multilayer which may entangle with each other and leads to higher viscosity. This effect of graphene continues as the temperature approaches to 100°C. At higher temperatures, entangled layers starts to open which results in less viscous behavior. As CNT has very small length ($<2\mu$ m) and other nanoparticles are spherical, viscosity increment is not substantial especially at higher shear rates which helps in dispersing the particles properly. Cobalt and alumina containing nano fluids have a tendency to give high viscosity at moderate temperatures but as the temperature increases to a larger value, the effect diminishes. High temperatures are more favorable for nanosilica and CNT addition to water as it gives better rheology to it. The study shows that nano fluid having nickel nanopowder demonstrate similar viscous behavior as that of pure water.



◆ Pure Water+0.6%nanosilica ■ Pure Water+0.6%alumina ● Pure Water+0.6%cobalt



■ Pure Water+0.6%graphene △ Pure Water+0.6%CNT O Pure Water

◆ Pure Water+0.6%nanosilica ■ Pure Water+0.6%alumina ● Pure Water+0.6%cobalt

A Pure Water+0.6%nickel



■ Pure Water+0.6%graphene △ Pure Water+0.6%CNT O Pure Water

◆ Pure Water+0.6%nanosilica ■ Pure Water+0.6%alumina ● Pure Water+0.6%cobalt

A Pure Water+0.6%nickel





◆ Pure Water+0.6%nanosilica ■ Pure Water+0.6%alumina ● Pure Water+0.6%cobalt

A Pure Water+0.6%nickel



Figure 5.1 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 30°C (Top) and 60°C (Bottom)



◆ Pure Water+0.6%nanosilica ■ Pure Water+0.6%alumina ● Pure Water+0.6%cobalt

Shaer Stress (mPa)

O Pure Water ■ Pure Water+0.6%graphene △ Pure Water+0.6%CNT

◆ Pure Water+0.6%nanosilica ■ Pure Water+0.6%alumina ● Pure Water+0.6%cobalt

A Pure Water+0.6%nickel



O Pure Water ■ Pure Water+0.6%graphene △ Pure Water+0.6%CNT

◆ Pure Water+0.6%nanosilica ■ Pure Water+0.6%alumina ● Pure Water+0.6%cobalt

A Pure Water+0.6%nickel



Figure 5.2 : Effect of 0.6 weight% Nanoparticles Addition in Pure Water at 100°C (Top) and 140°C (Bottom)

5.1.2 Base Fluid 1

The rheological behavior of nanoparticle addition to base fluid 1 (BF1) are analyzed in two ways. First way of analysis is to study the effect of different type of nanoparticle addition to BF1 by keeping the constant concentration at different temperatures. In order to see the behavior of a specific nanoparticle, another analysis is designed in which the effect of increase in concentration of nanoparticle is studied at different temperatures.

5.1.2.1 Effect of Type of Nanoparticle

In order to illustrate the effect of four different nanoparticles addition to BF1, the graphs of shear stress and viscosity are generated at different temperatures by keeping similar concentration of nanoparticle for comparison. Two concentrations (0.2% and 0.6% by weight) are selected during the study.

• <u>BF1 + 0.2% Nanoparticles</u>

Shear stress and viscosity results of BF1 with 0.2% of nanoparticles are shown in Figure 5.3 and Figure 5.4. As the fluid contains polymers, the behavior is similar to Non-Newtonian fluids with shear thinning effect but this effect is more pronounced with nanoparticle addition.

At 30°C and 60°C, nanofluid containing graphene shows better results in terms of viscosity as compared to other nanoparticles. It improves the yield point (YP) of BF1 which can be beneficial in hole cleaning. Both viscosity and shear stress get increased. The effect of graphene in BF1 at higher temperature (140°C) overlaps with BF1. Addition of graphene in BF1 results in relatively high viscosity than other nanoparticles due to the presence of multilayer structure of graphene. This phenomenon is limited when the temperatures are low. Layers starts to oscillate at higher temperature and the viscosity of fluid get back to its original level. High concentrations of graphene may resist this process and keep viscous behavior till much higher temperatures. Other viscosifiers can be added to aid graphene in improving yield point.

Nanosilica addition to BF1 also demonstrates better YP especially at temperatures up to 100°C. The effect of CNT in BF1 is almost similar to that of nanosilica in terms of giving similar level of yield point and plastic viscosity. The addition of alumina to BF1 does not illustrate significant change in the viscous behavior when the concentration of alumina is 0.2% by weight.

© BF1 ◆ BF1+0.2%graphene ■ BF1+0.2%CNT ● BF1+0.2%nanosilica ▲ BF1+0.2%alumina

© BF1 ◆ BF1+0.2%graphene ■ BF1+0.2%CNT ● BF1+0.2%nanosilica ▲ BF1+0.2%alumina





○ BF1 ◆ BF1+0.2%graphene ■ BF1+0.2%CNT ● BF1+0.2%nanosilica ▲ BF1+0.2%alumina

○ BF1 ◆ BF1+0.2%graphene ■ BF1+0.2%CNT ● BF1+0.2%nanosilica ▲ BF1+0.2%alumina



Figure 5.3 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)

⊙BF1 ◆BF1+0.2%graphene ■BF1+0.2%CNT ●BF1+0.2%nanosilica ▲BF1+0.2%alumina

© BF1 ◆ BF1+0.2%graphene ■ BF1+0.2%CNT ● BF1+0.2%nanosilica ▲ BF1+0.2%alumina





⊙BF1 ◆BF1+0.2%graphene ■BF1+0.2%CNT ●BF1+0.2%nanosilica ▲BF1+0.2%alumina

○ BF1 ◆ BF1+0.2%graphene ■ BF1+0.2%CNT ● BF1+0.2%nanosilica ▲ BF1+0.2%alumina



Figure 5.4 :Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)

• <u>BF1 + 0.6% Nanoparticles</u>

Figure 5.5 and Figure 5.6 illustrates the behavior of 0.6% concentration of nanoparticles in BF1 at four temperatures. The graphs show how shear stress and viscosity of nano drilling fluid are improved when nanoparticles are added to BF1. The viscous behavior of nanosilica in BF1 at 0.6% concentration is higher than other nanoparticles at all temperatures. One of the potential reasons can be a stable solution of nanosilica at higher concentration in the presence of polymers which keeps nanosilica well dispersed in the BF1. Except at 140°C, BF1 containing nanosilica initially follows an increasing trend in shear stress with shear rate. Then it decreases followed by another increasing manner. At 140°C, the behavior of shear stress at all shear rates is an increasing one.

Unlike to nanosilica, graphene addition to BF1 shows a linear increase on log-log plot at all temperature. The rheology of BF1 also gets improved and better yield points are obtained at all temperatures as compared to BF1. Shear thinning behavior is dominant at all temperatures with viscosity decreases as temperature gets increased. BF1 with 0.6% of CNT also demonstrates linear relationship between shear stress and shear rate on log-log plot at all temperatures. At all temperatures, shear stress and viscosity of BF1 containing CNT approaches towards the values of BF1 as the shear rate becomes high (1020s⁻¹). Alumina containing BF1 illustrate different behavior at all temperatures by decreasing the viscosity lower than the BF1. At higher concentration of alumina particles, potential coagulation leads to settling of particles which may also take other dispersed additives with itself. Therefore, the viscosity of the remaining fluid gets decreased.

By evaluating two different concentration of nanoparticles in BF1, it can be said that addition of graphene at 0.2wt% bring major improvement in the viscosity of BF1 as compared to other nanoparticles at that concentration specially at temperature below 100°C. At 0.6wt% concentration, performance of nanosilica over shadows other nanoparticles.

© BF1 ◆ BF1+0.6%graphene ■ BF1+0.6%CNT ● BF1+0.6%nanosilica ▲ BF1+0.6%alumina

© BF1 ◆ BF1+0.6%graphene ■ BF1+0.6%CNT ● BF1+0.6%nanosilica ▲ BF1+0.6%alumina





◎ BF1 ◆ BF1+0.6%graphene ■ BF1+0.6%CNT ● BF1+0.6%nanosilica ▲ BF1+0.6%alumina

O BF1 ◆ BF1+0.6%graphene ■ BF1+0.6%CNT ● BF1+0.6%nanosilica ▲ BF1+0.6%alumina



Figure 5.5 : Effect of 0.6 weight% Nanoparticles Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)



⊙ BF1 ◆ BF1+0.6%graphene ■ BF1+0.6%CNT ● BF1+0.6%nanosilica ▲ BF1+0.6%alumina ○ BF1 ◀

© BF1 ◆ BF1+0.6%graphene ■ BF1+0.6%CNT ● BF1+0.6%nanosilica ▲ BF1+0.6%alumina



○ BF1 ◆ BF1+0.6%graphene ■ BF1+0.6%CNT ● BF1+0.6%nanosilica ▲ BF1+0.6%alumina

O BF1 ◆ BF1+0.6%graphene ■ BF1+0.6%CNT ● BF1+0.6%nanosilica ▲ BF1+0.6%alumina



Figure 5.6 : Effect of 0.6 weight% Nanoparticles Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)

5.1.2.2 Effect of Concentration of Nanoparticle

Another study is done to evaluate the effect of increase in concentration of particular nanoparticle in BF1 at different conditions of temperature. In this study, the behavior of each nanoparticle having two concentrations (0.2% and 0.6%) is compared with the behavior of BF1, free of nanoparticles.

• <u>Graphene</u>

The concentration of graphene in BF1 are varied and its effects on rheology at different temperature are analyzed (Figure 5.7 to Figure 5.8). At 30°C and 60°C, both concentration of graphene in BF1 have similar curves of shear stress and viscosity. It is an important finding which can help in selecting proper concentration of graphene to improve viscosity. The behavior shifts towards a new side when the fluids are tested at 100°C and 140°C where 0.6% graphene addition by weight in BF1 results in high viscosity as compared to 0.2% concentration.

• <u>CNT</u>

Figure 5.9 and Figure 5.10 depicts the response of 0.2% and 0.6% of CNT addition to BF1. BF1 containing 0.6% CNT has higher viscosity than 0.2% at any shear rate except when the temperature of sample is 140°C where the effect of concentration of CNT is not prominent. The behavior is quite obvious as increase in concentration results in more dispersed solids in the fluid which cause more resistance between fluid layers on shearing.

• <u>Nanosilica</u>

The addition of nanosilica at 0.6% concentration level to BF1 clearly shows higher level of shear stress and viscosity at all temperatures as compared to 0.2% concentration level. The effect is shown in Figure 5.11 and Figure 5.12. The difference in behavior of two concentrations is more pronounced at low shear rates except when the sample temperature is maintained at 140°C.

• <u>Alumina</u>

As mentioned earlier that the behavior of BF1 containing alumina at all concentration is different from that of other particles, Figure 5.13 and Figure 5.14 demonstrate similar results of shear stress and viscosity. In the beginning, when the temperature is maintained at 30°C, the effect of increasing concentration of alumina in BF1 leads to decrease in viscous behavior of nano drilling fluid. The effect continues till the sample is tested at 100C. Then this trend weakens as temperature is increased to 140°C and the effect of concentration of rheology is no longer existed. The coagulation of alumina particles is responsible for the behavior.

In general, higher concentration of nanoparticles in drilling fluid leads to more viscous behavior as shown by nanosilica and CNT but particles like alumina can offer opposite behavior. It is also noticed that viscosity of graphene in BF1 does not depend heavily on its concentration.


Figure 5.7 : Effect of Graphene Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)



Figure 5.8 : Effect of Graphene Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)



Figure 5.9 : Effect of CNT Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)



Figure 5.10 : Effect of CNT Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)



Figure 5.11 : Effect of Nanosilica Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)



Figure 5.12 : Effect of Nanosilica Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)



Figure 5.13 : Effect of Alumina Addition in Base Fluid 1 at 30°C (Top) and 60°C (Bottom)



Figure 5.14 : Effect of Alumina Addition in Base Fluid 1 at 100°C (Top) and 140°C (Bottom)

5.1.3 Base Fluid 2

The study of rheological behavior of drilling fluid is repeated with BF2 and the effect of addition of nanoparticles is evaluated by comparing shear stress and viscosity results at different temperature. Similar concentrations (0.2% and 0.6% by weight) of four types of nanoparticles are analyzed in BF2 and graphs of shear stress and viscosity are compiled as a function of shear rate at different temperatures. Effect of type of nanoparticle as well as concentration of nanoparticles are studied which is covered in this section.

5.1.3.1 Effect of Type of Nanoparticle

BF2 is prepared with two concentrations (0.2% and 0.6%) of each nanoparticle and the results of shear stress and viscosity are compared with original BF2 containing no nanoparticles.

• <u>BF2+0.2% nanoparticles</u>

Figure 5.15 and Figure 5.16 illustrate the behavior of 0.2% concentration of nanoparticles in BF2 at four temperatures. The graphs show how shear stress and viscosity of nano drilling fluid are shifted when nanoparticles are added to BF2. All the nanoparticles alter the rheology of BF2 and transfer the shear stress and viscosity curves below the original curve of BF2.

Out of all nanoparticles, graphene addition causes a significant reduction in viscosity of drilling fluid. As the temperature increases from 30°C to 100°C, the difference of curves between BF2 and graphene added BF2 widens which is then followed by relatively smaller difference in the end at 140°C. Alumina addition to BF2 also decreases the viscous behavior of BF1 at all temperatures. Similar kind of behavior is shown by alumina as that of graphene when temperature varies from 30°C to 100°C. BF2 containing nanosilica also cause a decreasing effect in viscosity specially at 30°C, 60°C and 100°C but this decreasing effect vanishes as the shear rate approaches to higher values. At 140°C, it follows the shear stress and viscosity trends of BF1. CNT addition to BF2 also decreases the shear stress and viscosity of BF2 at all temperature. As the temperature increases from 30°C to 100°C, both shear stress and viscosity of BF1 with 0.2% CNT decreases proportionally.

A highly alkaline environment is one of the reasons behind this effect. In neutral or near neutral environments (as in BF1), nanoparticles have shown an increasing trend both in shear stress and viscosity as described in Section 5.1.2 High pH of drilling fluid creates such conditions in which polymers can react with other additives which are not likely to occur at low pH values. pH of drilling fluid depends upon the temperature. High temperature favors more generation of hydrogen ions in the fluid which leads to lower pH value. A less alkaline environment is more favorable for nanoparticles to act as viscosifier. Therefore, at higher temperature (140°C), the viscosity of nano drilling fluids begins to approach base fluid viscosity due to less alkaline environment.

© BF2 ◆ BF2+0.2%graphene ■ BF2+0.2%CNT ● BF2+0.2%nanosilica ▲ BF2+0.2%alumina

© BF2 ◆ BF2+0.2%graphene ■ BF2+0.2%CNT ● BF2+0.2%nanosilica ▲ BF2+0.2%alumina





○ BF2 ◆ BF2+0.2%graphene ■ BF2+0.2%CNT ● BF2+0.2%nanosilica ▲ BF2+0.2%alumina

○ BF2 ◆ BF2+0.2%graphene ■ BF2+0.2%CNT ● BF2+0.2%nanosilica ▲ BF2+0.2%alumina



Figure 5.15 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 2 at 30°C (Top) and 60°C (Bottom)

© BF2 ◆ BF2+0.2%graphene ■ BF2+0.2%CNT ● BF2+0.2%nanosilica ▲ BF2+0.2%alumina

© BF2 ◆ BF2+0.2%graphene ■ BF2+0.2%CNT ● BF2+0.2%nanosilica ▲ BF2+0.2%alumina





○ BF2 ◆ BF2+0.2%graphene ■ BF2+0.2%CNT ● BF2+0.2%nanosilica ▲ BF2+0.2%alumina

O BF2 ◆ BF2+0.2%graphene ■ BF2+0.2%CNT ● BF2+0.2%nanosilica ▲ BF2+0.2%alumina



Figure 5.16 : Effect of 0.2 weight% Nanoparticles Addition in Base Fluid 2 at 100°C (Top) and 140°C (Bottom)

• <u>BF2+0.6% nanoparticles</u>

The effect of 0.6% concentration of different nanoparticles on rheology is analyzed at different temperature which is shown in Figure 5.17 and Figure 5.18. Similar to 0.2% addition, concentration of 0.6% also cause less shear stress and low viscosity at approximately all shear rates as compared to the response of BF2 due to the reasons already mentioned in previous section.

At 30°C, graphene and alumina base fluids illustrate similar trend in terms of rheology. CNT and nanosilica initially reduces the viscosity of BF2 but as the shear rate increases, the viscosity of both nano fluids begins to approach the BF2 viscosity. At 60°C and 100°C, similar trends are shown by nanoparticles with BF2 containing alumina being the least viscous of all other fluids while BF2 having nanosilica is the highest among nano fluids. Viscosity of all the nano fluids is close to viscosity of BF2 at higher values of shear rates when the sample temperature is maintained at 140°C.

Due to high alkalinity of BF2, addition of nanoparticles in it results in reduction of viscosity. Low value of pH is suitable to get viscous effects in drilling fluid using nanoparticles. Other additives such as weighting material also have their influence in determining the overall performance of nano drilling fluid.

© BF2 ◆ BF2+0.6%graphene ■ BF2+0.6%CNT ● BF2+0.6%nanosilica ▲ BF2+0.6%alumina

© BF2 ◆ BF2+0.6%graphene ■ BF2+0.6%CNT ● BF2+0.6%nanosilica ▲ BF2+0.6%alumina





○ BF2 ◆ BF2+0.6%graphene ■ BF2+0.6%CNT ● BF2+0.6%nanosilica ▲ BF2+0.6%alumina

○ BF2 ◆ BF2+0.6%graphene ■ BF2+0.6%CNT ● BF2+0.6%nanosilica ▲ BF2+0.6%alumina



Figure 5.17 : Effect of 0.6 weight% Nanoparticles Addition in Base Fluid 2 at 30°C (Top) and 60°C (Bottom)

© BF2 ◆ BF2+0.6%graphene ■ BF2+0.6%CNT ● BF2+0.6%nanosilica ▲ BF2+0.6%alumina

© BF2 ◆ BF2+0.6%graphene ■ BF2+0.6%CNT ● BF2+0.6%nanosilica ▲ BF2+0.6%alumina



Figure 5.18 : Effect of 0.6 weight% Nanoparticles Addition in Base Fluid 2 at 100°C (Top) and 140°C (Bottom)

5.1.3.2 Effect of Concentration of Nanoparticle

In this study, the behavior of each nanoparticle having concentrations (0.2% and 0.6%) is compared with the behavior of BF2 containing no particles in it.

• <u>Graphene</u>

Figure 5.19 and Figure 5.20 depicts the response of 0.2% and 0.6% of graphene addition to BF2. Both concentrations cause reduction in BF2 viscosity. BF2 containing 0.6% graphene has higher viscosity than 0.2% at any shear rate irrespective of the temperature conditions due to presence of relatively large solids which offers more resistance between the layers and thus cause an increase in viscous behavior. The performance of graphene in drilling fluid is highly dependent on pH of drilling fluid.

• <u>CNT</u>

The concentration of CNT in BF2 is varied and its effects on rheology at different temperature are analyzed (Figure 5.21 to Figure 5.22). At all temperatures, increase in concentration from 0.2% to 0.6% does not make any significant difference on shear stress and viscosity as the rounded profile of CNT offers less resistance to shear the fluid. As the shear stress increases at particular temperature, the difference of results between BF2 and CNT fluid reduces.

• <u>Nanosilica</u>

The addition of nanosilica at 0.6% concentration level to BF2 clearly shows similar level of shear stress and viscosity at all temperatures as that of 0.2% concentration level. Addition of more spherical particles compensates the effect of high number of solids at 0.6% concentration as compared to 0.2%. The results are shown in Figure 5.23 and Figure 5.24. At low shear rates, both concentration cause reduction in viscosity of BF2.

• <u>Alumina</u>

Results of BF2 containing different concentration of alumina are compared with results of BF2 in Figure 5.25 and Figure 5.26. It is apparent from figures that 0.6% concentration leads to less viscous fluid as compared to fluid containing 0.2% alumina till the sample temperature is kept at 100°C. At 140°C, BF2 having 0.6% concentration cause an increase in viscosity than 0.2% concentrated fluid and this viscosity approaches to BF2 at higher shear rates.

Higher concentration of nanoparticles in BF2 can be used to improve the viscosity profile if the alkalinity of drilling fluid is maintained at lower level.



Figure 5.19 : Effect of Graphene Addition in Base Fluid 2 at 30°C (Top) and 60°C (Bottom)



Figure 5.20 : Effect of Graphene Addition in Base Fluid 2 at 100°C (Top) and 140°C (Bottom)



Figure 5.21 : Effect of CNT Addition in Base Fluid 2 at 30°C (Top) and 60°C (Bottom)



Figure 5.22 : Effect of CNT Addition in Base Fluid 2 at 100°C (Top) and 140°C (Bottom)



Figure 5.23 : Effect of Nanosilica Addition in Base Fluid 2 at 30°C (Top) and 60°C (Bottom)



Figure 5.24 : Effect of Nanosilica Addition in Base Fluid 2 at 100°C (Top) and 140°C (Bottom)



Figure 5.25 : Effect of Alumina Addition in Base Fluid 2 at 30°C (Top) and 60°C (Bottom)



Figure 5.26 : Effect of Alumina Addition in Base Fluid 2 at 100°C (Top) and 140°C (Bottom)

5.2 Tribometer Measurements

The tribological behavior of nano based fluid is studied through pin-on disk apparatus by determining the friction factor between ball and steel plate. Three base fluids are tested at different set of temperature with and without nanoparticles and lubrication behavior of each fluid is estimated. The friction factors obtained in the study are higher than those presented in Table 2.4 due to difference in experimental conditions and equipment limitations.

5.2.1 Pure Water

Figure 5.27 illustrates the behavior of pure water and nanoparticles addition in it at different temperatures. Minimum, maximum and mean friction factors are recorded at each temperature at the end of experiment with each type of fluid. Standard deviation is also given by the software to analyze the variation of friction factor during the experiment. Table 5.1 shows friction factor at four temperatures for different type nanoparticles added in the pure water at concentration of 0.6 weight%. Mean friction factor for water increase from 0.392 at 30°C to 0.503 at 60°C.

The effect of graphene addition to pure water at different temperature is shown in Figure 5.27. It can be clearly observed that the addition of graphene results in almost similar friction factor as that of pure water at all temperatures. CNT also performs similar to graphene and it leads to same lubrication behavior in pure water. Nanosilica, alumina and nickel give higher friction factor as compared to the pure water at all temperatures. Nanoparticles of cobalt show different results from the results of other particles. The graph in Figure 5.27 compares the performance of cobalt with pure water at all temperatures. At low temperatures, reduction of friction factor by cobalt in pure water is small but at 60°C the effect is very significant. The lubrication between ball and steel plate get improved when cobalt nanoparticles are added to pure water.

Results of pure water as base fluid show that friction factor depends upon temperature and it is generally increases with temperature. Viscosity reduction at higher temperature can be one of the mechanisms that can explain relationship between friction factor and temperature. Layered structure of graphene does not cause any reduction in friction while the cylindrical structure of muti-walled carbon nanotube helps in decreasing friction factor. Another possible explanation to this phenomenon can be better stability of pure water having CNT than that of having graphene. Among other nanoparticles, only cobalt addition to PW results in less friction factor due to its ball bearing effect between steel pin and disc.

In general, CNT and cobalt offer reduction in friction factor of pure water at concentration of 0.6wt%.

TEMPERATURE	PURE WATER	TYPE OF NANOPARTICLE							
(° C)		Graphene	NanoSilica	CNT	Alumina	Cobalt	Nickel		
30	0.392	0.403	0.429	0.378	0.427	0.368	0.426		
40	0.446	0.432	0.452	0.430	0.492	0.417	0.447		
50	0.446	0.459	0.499	0.441	0.540	0.429	0.496		
60	0.503	0.493	0.544	0.497	0.552	0.436	0.506		

 Table 5.1 : Mean Friction Factor with Pure Water



Figure 5.27 : Effect of Nanoparticles Addition in Pure Water

5.2.2 Base Fluid 1

Base fluid 1 (BF1) is also prepared and is brought under study for analyzing its tribological behavior at different temperature. Figure 5.28 depicts how friction factor of BF1 vary with the temperature when nanoparticles are added. It also manifests that the tribological properties of BF1 do not change very much with temperature. This serves as the base for comparison with effect of nanoparticle addition. A brief overview of nanoparticle addition to BF1 can be realized from Table 5.2. Four types of nanoparticle are analyzed in tribometer to determine the effect on friction factor. Two sets of concentration (0.2 % and 0.6%) are chosen for each nanoparticle to observe the consequence of concentration increment.

Figure 5.28 also depicts the lubrication behavior of graphene addition to BF1 at different temperatures. At 30°C, reduction of friction factor is observed with both 0.2% as well as 0.6% of graphene. Higher temperatures result in high friction factor. With the increase in graphene concentration, friction factor remains relatively at the same level. Therefore, friction factor of nanofluid containing graphene does not depend on its concentration. With the addition of nanosilica, CNT and alumina to BF1, increasing trend of friction factor is observed with respect to base fluid at all temperatures except for the BF1 containing 0.2wt% CNT at 30°C. This can be explained by the stability of the fluid. Due to settling of nanoparticles on disc, the frictional resistance between ball and plate increases. Comparison of results of graphene with

other nanoparticles at 0.6 wt% reveals that graphene cause lesser increase in friction factor at all temperature.

It can be stated as conclusion that addition of nanoparticles in BF1 does not cause reduction in friction factor.

		CONCENTRATION AND TYPE OF NANOPARTICLE							
TEMPERARTURE (°C)	BF 1	Graphene		NanoSilica		CNT		Alumina	
		0.2	0.6	0.2	0.6	0.2	0.6	0.2	0.6
		wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
30	0.333	0.293	0.323	0.344	0.325	0.307	0.378	0.338	0.354
40	0.309	0.332	0.327	0.362	0.356	0.329	0.384	0.356	0.385
50	0.311	0.348	0.336	0.360	0.378	0.333	0.399	0.364	0.431
60	0.310	0.365	0.356	0.360	0.395	0.326	0.396	0.374	0.442

 Table 5.2 : Mean Friction Factor with Base Fluid 1





Figure 5.28 : Effect of Nanoparticles Addition in Base Fluid 1

5.2.3 Base Fluid 2

Similar to BF1, base fluid 2 (BF2) is prepared carefully in the laboratory by mixing the chemicals as mentioned in the Section 4.1. Friction factor results obtained from tribometer with BF2 as well as its nano drilling fluid are illustrated in Figure 5.29. As this base fluid consists of relatively large amount of chemicals as compared to BF1, therefore, friction factors are higher than previously obtained. Similar concentrations of each nanoparticle are tested with BF2 and tribological behavior is studied at different temperatures. Table 5.3 summarizes the friction factor of nano-based drilling fluid when each nanoparticle is added to BF2. Figure 5.29, showing the results for graphene addition, prove that lubrication effect of drilling fluid can be improved by adding graphene to it. The effect is more pronounced at higher concentration (0.6%) of graphene at all temperatures. This effect can be explained by formation of smear film by multilayered structure of graphene at high pH in the presence of other chemical. The film provides less frictional resistance between steel ball and plate. The performance of nano based fluid using nanosilica is somewhat different from graphene. At low concentration, friction factors are relatively higher but as the concentration increases the friction factor decreases (Figure 5.29). Nanosilica particles generate ball bearing effect during friction factor measurement and thus lead to relatively low values. BF2 containing CNT provide excellent lubrication between ball and steel plate and gives less friction factors at all temperature as compared to BF2 without nanoparticles. CNT comprises of graphene sheets as its wall forming material which is then rolled to form nanotubes. Due to rolled outer profile, CNT in BF2 offers less friction factor. Alumina addition to the BF2 yield low friction factor at 0.2 wt% concentrations but high friction factor at 0.6wt% concentration.

Unlike to PW and BF1, the performance of following nanoparticles in BF2 is quite promising in terms of friction factor reduction.

- 0.6% graphene
- 0.6% nanosilica
- 0.2% CNT
- 0.6% CNT
- 0.2% alumina

TEMPERARTURE (°C)	BF 2	CONCENTRATION AND TYPE OF NANOPARTICLE							
		Graphene		NanoSilica		CNT		Alumina	
		0.2	0.6	0.2	0.6	0.2	0.6	0.2	0.6
		wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
30	0.408	0.438	0.409	0.498	0.353	0.373	0.331	0.385	0.449
40	0.438	0.456	0.407	0.531	0.397	0.379	0.373	0.425	0.5
50	0.472	0.465	0.365	0.531	0.415	0.365	0.326	0.446	0.522
60	0.502	0.492	0.456	0.504	0.403	0.338	0.338	0.435	0.532

Table 5.3 : Mean Friction Factor with Base Fluid 2



Figure 5.29 : Effect of Nanoparticles Addition in Base Fluid 2

5.3 Density Measurements

Density of all the base fluids and nano drilling fluids are measured through Anton Paar's density meter. The measured data is then studied with respect to each base fluid and the effect of nanoparticles is analyzed with different concentrations.

5.3.1 Pure Water

Figure 5.30 illustrates the effect of nanoparticles addition to pure water (PW) in terms of density of fluid at 20°C. It is apparent from the figure that pure water containing nanoparticle is slightly denser than pure water. Nanosilica addition to PW at concentration of 0.6% by weight gives the highest density followed by alumina and nickel. PW containing cobalt and graphene demonstrates similar level of density while addition of CNT in PW does not affect the density of it.



Figure 5.30 : Effect of Nanoparticles Addition on Density of Pure Water

5.3.2 Base Fluid 1

Density results of BF1 with and without nanoparticles are shown in Figure 5.31. All the nano fluid containing 0.2% nanoparticles do not change the density of BF1. When the concentration of each nanoparticle is modified from 0.2% to 0.6%, the density of nano fluids shift towards a higher value with fluid containing 0.6% CNT leading all the other nano fluids.



Figure 5.31 : Effect of Nanoparticles Addition on Density of Base Fluid 1

5.3.3 Base Fluid 2

BF2 is also studied through similar density meter as that of PW and BF2, and the measured data is illustrated in Figure 5.32. It is evident from the figure that the density level of BF2 is maintained at approximately same level even after the addition of nanoparticles at all concentrations except for graphene addition which leads to a reduction in viscosity as its concentration in BF2 increases.



Figure 5.32 : Effect of Nanoparticles Addition on Density of Base Fluid 2

Addition of nanoparticles in all base fluids causes slight increase in density as the nanoparticles size and concentration is very small except for the graphene in BF2 which shows a reduced density. Therefore, nanoparticles do not have a significant tendency to make drilling fluid denser. Due to this, weighting material such as barite or calcium carbonate (as in BF2) must be used in drilling fluid in order to control and modify the density.

5.4 pH Measurements

The measurement of pH is carried out in the instrument lab with the help of pH meter. pH of all the base fluids and nano fluids is measured at 25°C by immersing the electrode of pH meter in specific volume of sample fluid.

5.4.1 Pure Water

Measured values of pH for pure water with and without nanoparticles are shown in Figure 5.33. All the nano fluids cause reduction in pH of PW at nanoparticles concentration of 0.6% except pure water containing cobalt and nickel. Maximum reduction in pH is experienced when graphene is added to water.



Figure 5.33 : Effect of Nanoparticles Addition on pH of Pure Water

5.4.2 Base Fluid 1

Figure 5.34 illustrates the effect of pH due to nanoparticles addition into BF1. It is apparent from the figure that pH follows a decreasing trend when concentration of graphene and nanosilica increases from 0.2% to 0.6% by weight. Addition of CNT and alumina particles into BF1 results in higher pH than that of original BF1.



Figure 5.34 : Effect of Nanoparticles Addition on pH of Base Fluid 1

5.4.3 Base Fluid 2

pH measurements for BF2 and its relevant nano drilling fluids are shown in Figure 5.35. As the BF2 contains NaOH, pH values are towards higher side. All the nano drilling fluids illustrate reduced pH as compared to BF2 at all concentrations. Figure 5.35 clearly shows that graphene and nanosilica addition reduces the pH as the concentration increases. This decreasing trend can also be observed for alumina but it is not as much prominent as it is for graphene and nanosilica. BF2 containing CNT does not show any significant change in pH when the concentration is increased from 0.2% to 0.6% by weight.



Figure 5.35 : Effect of Nanoparticles Addition on pH of Base Fluid 2

pH level of drilling fluid is very vital as it determines the performance of additives and polymers. Normally field waters that are used to prepare drilling mud contains large amount of calcium and magnesium which creates hardness in water. Certain chemicals such as soda ash are added to precipitate ions of calcium and thereby increase the pH. High pH environments result in desired chemical reaction between drilling fluid additives and provide sufficient yield. pH study during the project shows that all the selected nanoparticles cause reduction in pH both in neutral (PW and BF1) as well as alkaline environment (BF2). Therefore proper amount of soda ash or any other additive for pH enhancement must be selected so that pH reduction due to nanoparticles in drilling fluid can be compensated by addition of soda ash or other additives.

6 Modeling Work

6.1 Introduction

This chapter deals with the application of experimental results in analyzing different models for studying important parameters involved in drilling operations. The study comprises of temperature, viscosity, friction factor, torque and drag models. The work flow of modeling begins with the selection of base fluid along with those nanoparticles which show reduction in friction factor during tribometer experiment. Afterwards, viscosity and friction factor models are generated for a wellbore case study as a function of temperature based on the experimental results. These models are then introduced to software program to generate viscosity and friction factor profile along the well bore as function of well bore temperature which is already determined based on temperature model. Based on friction factor profile, torque and drag results for the best nano drilling fluid is obtained which is later compared with base fluid offers less friction factor. In addition to this, conclusions on bit cooling are also drawn by comparing the temperature profiles of base fluid and nano drilling fluid.

In this chapter, a brief introduction of each model is presented and the assumptions on which it is based on. The details of each model can be accessed by using the provided references. This chapter focuses more on the discussion of results obtained from each model for BF2 as well nano drilling fluid. The results and discussion of each model are provided under the respective model section in this chapter.

6.2 Selection of Base Fluid and Nanoparticles

In order to develop models, base fluid and nanoparticles have to be selected at first. As the experimental analysis consist of three base fluids along with multiple nanoparticles at different concentrations, a screening criteria is developed for selecting from the available fluids. Some nanoparticles in Pure water (PW) have shown reduction in friction factor which is promising from torque and drag point of view. But the rheological behavior of these fluids does not have a capability to perform other functions of drilling fluid in the well such as cutting transport. Addition of nanoparticles in BF1 results in high friction factors as compared to BF1 which can increase torque and drag in the well. Unlike to PW and BF1, the behavior of few nanoparticles in BF2 is quite promising in terms of friction factor and viscosity. Therefore, following nano drilling fluids are selected for the further study.

- BF2 with 0.6% graphene
- BF2 with 0.6% nanosilica
- BF2 with 0.2% CNT
- BF2 with 0.6% CNT
- BF2 with 0.2% alumina

6.3 Case Study

Implementation and demonstration of models are performed in a case study in which a well is constructed on MATLAB. All the necessary well data is being placed as an input such as well diameter, pipe diameter, wellbore survey. In addition to this, Earth's surface temperature and geothermal gradient are also defined. Different properties of pipe, formation and base fluid are also inserted in the beginning such as thermal conductivity, specific heat, density and viscosity at surface. Viscosity and density of all the selected nano drilling fluids at surface conditions are also defined along with the base fluid. Circulation rate and time are chosen at an optimum level which is based on field experience.

6.4 Temperature Model

Holmes and Swift developed solution for calculating circulation temperature based on steady state heat transfer [62]. This model is implemented during the case study to determine temperature profiles inside the drill pipe and annulus with BF3 as well as nano drilling fluids. The model is highly sensitive to various parameters such as thermal conductivity and specific heat of formation, geothermal gradient, circulation rate and time, and drilling fluid properties. Therefore, a though study is carried out to select optimum values of these parameters.

Thermal conductivity and specific heat of nano drilling fluid have not been determined in currently available literature especially for selected nanoparticles in the base fluid used in this study. But it has been noticed by different researchers that addition of nanoparticle to fluid improve its heat transfer properties [63] [64] [65]. This is due to the high surface area of nanoparticles which aid in heat transfer. In the study, thermal conductivity of nano drilling is taken at slightly higher level than BF2 as no experimental data for thermal conductivity is available. Similar assumption is made for specific heat in which its value for nano drilling fluid is kept at slightly lower level as compared to BF2's specific heat. Both of these properties are kept at similar level for all the nanoparticles so that the effect of other experimentally measured properties such as density and surface viscosity can be evaluated.

The results of temperature profiles are shown in Figure 6.1 and Figure 6.2. Temperature profile inside drill pipe (Figure 6.1) presents a comparison of BF2 with other nano drilling fluids along with the geothermal gradient. Figure 6.1 shows that the well has been drilled in an area of normal geothermal gradient. Temperature of each fluid is highly dependent on heat transfer from formation. This heat transfer depends on thermal conductivity and specific heat of formation as well as fluid. It can be easily observed in the Figure 6.1 that nano drilling fluids have high temperature as compared to BF2 in down hole conditions. This is due their high thermal conductivity and lower specific heat than that of BF2. These properties allow nano drilling fluid to raise its temperature swiftly. BF2 containing 0.6wt% of graphene offers the highest temperature at down hole conditions due to its low viscosity at surface conditions. Other nano fluids also show better temperature profile as compared to BF2 but the results of graphene addition is the most prominent one.

Similar results of temperature profile can be observed in the annulus when the fluid is moving from bit towards the surface (Figure 6.2). All the fluids begin to heat up as formation is closer to them in the annulus which raises the temperature faster. Nano drilling fluids also show high temperature profile in the annulus as well due to their thermal properties specially the BF2 containing 0.6wt% graphene. All the fluids transfers heat to the shallower formation, casing and drill pipe during their movement in annulus. As nano drilling fluid has an ability to transfer heat quickly as compared to BF2, lower temperatures of nano drilling fluid can be observed at shallower depths particularly with graphene added fluid. Enhanced heat transfer characteristic of nano drilling fluid can help in bit cooling which leads to better durability of bit especially in high temperature and geothermal wells.


Figure 6.1 : Temperature Profile inside Drill Pipe



Figure 6.2 : Temperature Profile inside Annulus

6.5 Viscosity Model

Viscosity Models for BF2 and other nano drilling fluids are generated by using viscosity results from Modular Compact Rheometer (MCR) at different temperatures. The results of viscosity at four temperatures (30°C, 60°C, 100°C and 140°C) are used at a particular shear rate and linear regression analysis is applied to create a model. Table 6.1 shows viscosity model (μ) of each drilling fluid as function of well bore temperature(T).

FLUIDS	VISCOSITY MODELS
BF2	$\mu = -0.176T + 31.43$
BF2+0.6 wt% graphene	$\mu = -0.136T + 23.75$
BF2+0.6 wt% nanosilica	$\mu = -0.233T + 37.88$
BF2+0.2 wt% CNT	$\mu = -0.209T + 33.55$
BF2+0.6 wt% CNT	$\mu = -0.230T + 37.12$
BF2+0.2 wt% alumina	$\mu = -0.161T + 26.40$

 Table 6.1 : Viscosity Models for different Fluids

The models are then used to create viscosity profile inside the drill string and in the annulus which are shown in Figure 6.3 and Figure 6.4 respectively. Viscosity of all the fluids inside the drill pipe decreases as the fluid moves towards the bit due to an increasing fluid temperature (Figure 6.3). The behavior of nano drilling fluid is controlled by the slope and intercept of viscosity model which is based on experiments performed in the earlier part of study. 0.6% graphene and 0.2% alumina added fluids offer less viscous behavior as compared to BF2 where as fluids containing 0.6% CNT and 0.6% nanosilica show high viscosity inside the drill pipe.

Since the fluid initially heats up in the annulus, viscosity of each fluid presents a decreasing trend which is then followed by increase in viscosity as the fluid cools down when it moves towards shallower depths (Figure 6.4). High viscosity of nano drilling fluid containing 0.6% CNT and 0.6% nanosilica can result in better cutting transport than using BF2 as drilling fluid. The models state that viscosity of these fluids is more sensitive to temperature as compared to other nano drilling fluid. This behavior can also be noticed by analyzing the difference of surface and down hole viscosity.



Figure 6.3 : Viscosity Profile inside Drill Pipe



Figure 6.4 : Viscosity Profile inside Annulus

6.6 Friction Factor Model

Tribometer results of BF2 and nano drilling fluids are used to generate friction factor model as a function of temperature. It has been stated earlier that drilling fluid offer high friction factor at higher temperatures. Similar nature of results has been obtained from the tribometer experiments in which friction factor of some nano drilling fluid are strongly dependent on temperature. Table 6.2 shows the friction factor (μ) model as a function of temperature (T) obtained from linear regression analysis of experimental values.

FLUIDS	FRICTION FACTOR MODELS
BF2	$\mu = 0.00316T + 0.31280$
BF2+0.6 wt% graphene	$\mu = 0.00169T + 0.35064$
BF2+0.6 wt% nanosilica	$\mu = 0.00168T + 0.31640$
BF2+0.2 wt% CNT	$\mu = -0.00040T + 0.38833$
BF2+0.6 wt% CNT	$\mu = -0.00026T + 0.35370$
BF2+0.2 wt% alumina	$\mu = 0.00171T + 0.34580$

 Table 6.2 : Frcition Factor Models for different Fluids

Figure 6.5 presents friction factor profile along the annulus in the presence of BF2 as well as nano drilling fluids. As mentioned earlier that the selection of nano drilling fluid for case study is done on the basis of reduced friction factor, it is also translated by the model and can be observed in the Figure 6.5. Friction factor between contacting surfaces in the well bore is much lower in the presence of nano drilling fluids than BF2. As the fluids move up in the annulus, friction factor increases due to high fluid temperature which is because of larger heat transfer from formation. This trend of increase in friction factor does not last for long time and it is then followed by decrease in friction factor as the fluids become cooler at shallow depths.

Highest reduction in friction factor is experienced when the well is circulated with BF2 containing 0.6wt% of CNT. The graph also shows that friction factor model of 0.6wt% CNT added BF2 does not depend highly on fluid temperature in the annulus. This is extremely good for reducing torque and drag in high temperature and geothermal wells which offer extremely high bottom hole temperatures during drilling. The friction factor with 0.2wt% CNT added BF2 also have lower values than BF2. Out of remaining nano fluids, BF2 containing 0.6% of nanosilica demonstrate least friction factor and its profile is highly dependent on temperature. Friction factor, offered by graphene and alumina added BF2, are very similar to each other.



Figure 6.5 : Fricton Factor Profile

6.7 Torque and Drag Model

A three dimensional torque and drag model is implemented in the study which was developed by Aadnoy et. al [66]. The model determines torque and drag values by using different mathematical expressions for vertical, inclined and curved sections of the well. In order to identify vertical, inclined and curved sections, a DLS filter is introduced so that section identification can be done before proceeding to the calculation of torque and drag. Both torque and drag are studied in static as well as dynamic conditions such as hoisting and lowering of pipe. In addition to this, rotation of drill string is also introduced during lowering and hoisting operations to see the effects of combined motion on torque and drag. All the calculations of torque and drag are carried out using temperature dependent friction factor of each fluid instead of using a constant one. In this manner, model is made more reliable as it simulates the experimental observation.

BF2 containing 0.6wt% of CNT show better viscous behavior and reduced friction factor as compared to BF2 as well as other nano drilling fluids. Therefore, the study of torque and drag is carried out when the well is filled with 0.6wt% CNT added BF2 and then the results are compared with BF2. Torque and Drag results using BF2 as drilling fluid are shown in Figure 6.6 and Figure 6.7 where as for BF2 containing 0.6wt% CNT in Figure 6.8 and Figure 6.9.

A closer look on both figure 6.6 and Figure 6.8 can elaborate the significance of using nano drilling fluid in drag reduction. As the BF2 offers higher friction factor than CNT added based fluid, the drag difference during hoisting and lowering, with static load at particular depth is bigger than that of BF2 containing CNT. Drag during hoisting in the presence of CNT added BF2 is smaller as compared to the hoisting drag of BF2. For example, hoisting drag with nano drilling fluid at 1000m is 1155kN which has a value of 1200kN with BF2 without nanoparticle. Similar behavior can be observed for hoisting drag with combined motion.

Torque reduction, by using BF2 having 0.6wt%CNT as drilling fluid, can be confirmed by comparing the results of torque in Figure 6.7 and Figure 6.9 for different types of operations. Surface torque during static, hoisting and lowering operations is reduced by more than 20% when the well is filled with 0.6wt%CNT added BF2 instead of simple BF2. Similar trend can be seen at all depths as torque of drill string is lowered significantly in the well by nano drilling fluids.



Figure 6.6 : Drag Force in the Well with BF2



Figure 6.7 : Torque in the well with BF2



Figure 6.8 : Drag Force in the Well with 0.6%CNT in BF2



Figure 6.9 : Torque in the Well with 0.6%CNT in BF2

7 Conclusions and Recommendations

7.1 Conclusions

This study explores an innovative way of making the most of drilling fluid by adding nanoparticles in it. By studying rheological and tribological behavior of nano drilling fluid along with modeling work, following conclusions can be drawn,

- The behavior of nano drilling fluid is shear thinning.
- Viscosity of drilling fluid containing nanoparticles decreases with temperature.
- The viscous behavior of water can be improved by the addition of graphene at all temperatures.
- Presence of polymers has its impact on the performance of nanoparticles in drilling fluid such as graphene and nanosilica especially at low temperatures because each polymer has its maximum temperature range which defines its effective working window in drilling fluid.
- Multilayered structure of graphene can aid drilling fluid in improving its rheology and, thereby cutting transport efficiency. This improvement of rheology is strong function of its concentration, polymer content, down hole temperature and pH of drilling fluid.
- Nanoparticles contribute significantly in the viscosity of drilling fluid at low values of pH.
- pH of nano drilling fluid depends upon its temperature. High temperature weakens the alkaline nature of nano drilling fluid.
- Stability of nanoparticles in drilling fluid is very important in determining its rheological and tribological properties.
- High pumping rates are suitable for better dispersion of nanoparticles in drilling fluid and can be used to control its viscosity.
- Density of drilling fluid does not change noticeably with the addition of nanoparticles as the concentrations are kept at lower level.
- Friction factor offered by nano drilling fluid depends upon temperature.
- Nano drilling fluid can show reduction in friction factor provided that the concentration, polymer composition and pH are maintained at optimum level.
- In water based drilling fluid containing polymer at high pH, 0.6% of CNT by weight offer significantly reduced friction factor which leads to lower torque and drag.
- This fluid also has sufficient viscosity which can aid in transporting cutting from bottom to surface.
- Enhanced thermal conductivity of nano drilling fluid can help in cooling bit and results in longer bit life especially in high temperature well as the damaged such as heat checking can be avoided.

7.2 Recommendations for Future Work

Various aspects of nano drilling fluid can be investigated. Few recommendations are stated below which can be used to find other benefits of using nano drilling fluids over conventional fluids.

- Gel strength, filtration and filter cake thickness should be studied for nano drilling fluids.
- Stability of nanoparticles should be studied with the help some chemicals such as surfactants.
- Rheological and tribological experiments should be conducted at high pressure so that results become more reliable.
- Experiments should be performed to investigate thermal conductivity and specific heat of nano drilling fluid.

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