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CHEMICAL ENGINEERING | RESEARCH ARTICLE

Optimizing the rheological properties of silica nano-modified bentonite mud using overlaid contour plot and estimation of maximum or upper shear stress limit

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Abstract: An optimization based statistical (response surface) approach was used to evaluate the rheological properties of bentonite mud treated with silica nanoparticles. The overlaid contour plot established the feasible region for the various factor settings from multiple regression equations. The steepest method was used to further determine the optimal factor settings for minimum rheological properties and this was established at 6.3 wt.% bentonite content and 0.94 wt.% silica nanoparticles. The rheological properties of the bentonite mud containing and without silica nanoparticles was evaluated using a Hyperbolic (new) model and related with other oil industry based models: Herschel Bulkley, Sisko, Casson. The hyperbolic rheological model estimated the rheological behaviour of the nano-modified mud satisfactorily while also predicting a shear stress limit for the nano-modified mud. The maximum shear stress limit values for 6.3, 13 and 15 wt.% mud were 14.59, 61.74 and 107.4 Pa respectively. Upper shear stress values obtained from a 1.5 wt.% silica

Richard O. Afolabi

ABOUT THE AUTHORS

Mr Richard O. Afolabi is currently on his doctorate programme in Covenant University, Ota, Nigeria. Mr. Afolabi's research interests broadly encompasses the development of nanomaterials for application relevant to the oil and gas industry.

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Dr Vincent E. Efeovbokhan has varied experience spanning field operations and academia. His areas of core competence include alternative energy-biodiesel; product formulations (De-emulsifiers for crude oil treatment); environmental pollution control (bio-remediation).

Dr Oluwatosin J. Rotimi is an earth scientist with varied experience spanning Africa and the Far East. His areas of core competence include Seismic modelling (structural and stratigraphic), Sequence stratigraphy, Geostatistics, Environmental Geophysics and Petrophysics.

PUBLIC INTEREST STATEMENT

Oil and gas exploration and development is gradually shifting from onshore to deep offshore, which are under extreme environmental conditions. Drilling operations represents a critical part of oil and gas development hence the need to improve the rheological properties of the major ingredient of drilling operation, which is the drilling mud. Nanotechnology has provided the needed platform in improving drilling mud performance in deep offshore conditions. In this research work done, silica nanoparticles were used as additives in drilling mud and its rheological properties studied. The rheological behaviour was studied using conventional oil and gas models and this was compared with a new hyperbolic model. The hyperbolic model was able to go a step further and predict a new rheological property, which is the maximum or upper shear stress limit for drilling muds.

nanoparticle modified 6.3, 13 and 15 wt.% bentonite mud were 22.27, 72.62 and 171.3 Pa respectively, which represents an increment of 34.5 to 37.4% in the upper limit of shear stress. The effect of silica nanoparticles on the upper shear stress limit was quantified using a response surface design.

Subjects: Materials Science; Nanoscience & Nanotechnology; Chemical Engineering

Keywords: bentonite clays; silica nanoparticles; rheology; optimization; shear stress; contour plots; drilling mud; hyperbolic model

1. Introduction

Current research trends in most fields of study have shown a gradual shift to nanotechnology. In other words, the world is scaling down with respect to technological innovations and inventions and this has developed research interest in this area of technology, as it is the current domain for miniaturization with respect to scientific advancement. The oil and gas industry is not left out from this advancement as different areas from exploration, drilling to development and production have witnessed gradual research into the application of nanotechnology (Hoelscher, Stefano, Riley, & Young, 2012; Zakaria, Husein, & Hareland, 2012). Drilling operation is necessary for the confirmation of oil and gas beneath the earth surface and a major important ingredient in this operation is the drilling fluid which is often called drilling mud (Mahmoud, Nasr El-Din, Vryzas, & Kelessidis, 2016; Sehly et al., 2015). The use of nanoparticles as additives in bentonite drilling mud formulation is still relatively new in the petroleum industry. The addition of nanoparticles in the bentonite mud can be made into the desired viscosity and fluid loss attributes with varying amount of bentonite. This makes a good case for drilling mud formulation due to their ability to modify rheological attributes and fluid loss characteristics of the mud. Besides this, there has been no reported environmental impact of nanoparticle modified drilling mud. The use of nanoparticle for Water Based Muds (WBMs) in shale formations have shown how effective nanoparticles are in preventing fluid loss due to their small size (Hoelscher et al., 2012; Jung, Zhang, Chenevert, & Sharma, 2013; Sadeghalvaad & Sabbaghi, 2015; Zakaria et al., 2012).

2. Literature review

The flow characteristics of drilling fluids is one of the major influencing factor in the design, optimization and construction of oil and gas wells . The use of nanoparticles in drilling mud formulation has been reported to improve wellbore quality of shale formations by preventing the infiltration of mud containing water into the formation. This is achieved through its small size and large surface area. Nanoparticles can also influence rheological and thixotropic properties for drilling mud application. Hole cleaning often requires the removal of drilled cuttings and this is often done by passing the drilling mud through the drill string down to the drill bit where it exits into the hole and lifts the cuttings through the annulus to the surface. (Jung et al., 2013; Sadeghalvaad & Sabbaghi, 2015; Zakaria et al., 2012). Wyoming bentonite clays which represent the major ingredient in the formulation of drilling muds has been used over the years in performing functions such as the transport of drilled rock cuttings from the reservoir to the surface, preventing the loss of fluid into reservoir formations through the formation of filter cake on the reservoir rock surface, maintaining adequate hydrostatic pressure against formation pore pressure and erosion of reservoir rock during drilling operation. The presence of bentonite and other additives are essentially used to control and modify fluid loss characteristics and rheological properties such as yield stress, apparent viscosity, gel strength, plastic viscosity and the maximum shear stress limit. The higher limit of the shear stress or upper shear stress limit of the bentonite-based mud is an indication of how well the drilling mud erodes the rock formation. The lower limit of the shear stress or yield stress that can be created by the bentonite-based mud is a measure of resistance to flow or stress needed to initiate flow of the mud. This property better represents the pumpability of the mud.

Drilling muds exhibit non-Newtonian rheological behaviour and do not obey the direct relationship of rheological behaviour involving shear stress/shear rate. Restrictions exist on rheological models, which can explain the rheology of bentonite based muds as it relates to the prediction of the maximum shear stress tolerance (Vipulanandan & Mohammed, 2014). The current rheological models (Bingham Plastic Model, Power Model, etc.) enables viscosity and rheology data from rheological analysis to be fitted. The rheological properties obtained from these models tend to differ depending on the choice of model. The upper or maximum value for shear stress of a bentonite-based mud is influenced by the components of the bentonite-based mud. These models accepted in the oil industry cannot predict values for the upper limit of shear stress produced of a bentonite based mud. The upper limit of the shear stress of the bentonite based drilling mud represents its erosion potential.

Shear thinning fluids such as drilling muds are generally known to exhibit a rheological association, which mainly is non-linear, and have a maximum shear stress (upper shear stress). Other engineering applications involving the use of shear thinning fluids have been reported to be modelled with a hyperbolic relationship. Vipulanandan, Raheem, Basirat, and Mohammed (2014) used an hyperbolic model in investigating the formation of filter cake and fluid loss under High Temperature and High Pressure (HTHP) conditions. While the hyperbolic model used to predict the maximum limit for shear stress in a modified bentonite mud using polymer as additive, it can be extended to relate bentonite drilling mud rheology for other types of additives used for drilling mud in order to predict the the maximum shear stress limit.

The overall aim of the research include investigating rheological changes in the properties of a drilling mud containing silica nanoparticles. This is broken down into the following objectives:

(i) To examine the rheological properties of the silica treated bentonite mud and determine trade off optimal values using overlaid contour plots and steep method analysis.

(ii) To establish the rheological behaviour of the silica modified bentonite mud containing different silica nanoparticles amounts by means of a hyperbolic relationship and relate this model with other non-linear rheological models.

(iii) To statistically characterize the effect of silica nanoparticles on the upper limit of shear stress of the bentonite mud as determined using the hyperbolic model in (ii).

3. Materials and method

3.1. Materials

Silica nanoparticles (Size: 50 nm ± 4 nm, Appearance: white powder, Surface Area (TEM): 60.2 m^2/g, Purity: 99.8%) and bentonite clay were purchased from Equilab Solutions in Nigeria. The bentonite material was analysed based on the American Petroleum Institute (API) requirement (13A) for drilling mud materials (Table 1). Varying amount of bentonite (6.3, 13 and 15% by weight) was thoroughly mixed in deionised water using a Hamilton beach mixer. Modification of the bentonite mud with the silica nanoparticles was done by adding 0.5, 1 and 1.5 wt.% of the nanoparticles to each of the prepared mud containing 6.3, 13 and 15 wt.% bentonite respectively. The prepared drilling muds were allowed to age for 16 h before rheological analysis were carried out. The rheological characteristics of the treated drilling muds were compared with a base case sample (containing no silica nanoparticles).

3.2. Rheological measurements

The rheological properties and flow characteristics (rheological relationship) of the treated bentonite mud was investigated using a OFITE Model 800 Viscometer with 8 precisely regulated speed (in RPM: 3 (Gel), 6, 30, 60, 100, 200, 300 and 600 RPM). The viscometer was calibrated according to the API recommended practice 13B-1 and 13B-2 respectively. The calibrating instrument from OFITE was used to enable the calibration procedure. The speed was altered with a regulator knob and the dial readings were shown on a light enlarged dial. The Dial Readings (DR) and RPM values were converted to shear stress and shear rate values using the manufacturer's specification (Equations (1) and (2)) for the particular rotor bob used (Rotor-Bob Combination: R1B1):

Table 1. Bentonite physical requirements

Requirements	Specification	Mud sample
Viscometer dial reading at 600 RPM*	30, minimum	30
Viscometer dial reading at 300 RPM	–	23
Plastic viscosity	–	7
Yield point	–	16
Yield point/Plastic viscosity ratio*	3, maximum	2.29
Mud weight	–	8.6 ppg
Specific gravity	–	1.3
Filtrate volume*	15 cm^3, maximum	14 cm^3

*API requirements.

$$1\ \text{RPM} = 1.7023\ \text{s}^{-1}\ \text{RPM}^{-1} \tag{1}$$

$$1\text{DR} = 1.065\frac{Ib}{100ft^2} = 0.5107\ \text{Pa} \tag{2}$$

Based on the API specification 13A, Equations (3) to (5) was used in estimating the rheological attributes of the prepared drilling mud.

$$\text{Plastic viscosity (PV), cp} = \theta_{600} - \theta_{300} \tag{3}$$

$$\text{Yield point (YP)}, \frac{Ib}{100ft^2} = \theta_{300} - \text{PV} \tag{4}$$

$$\text{Apparent viscosity (AV), cp} = \theta_{600}/2 \tag{5}$$

θ_{600} and θ_{300} are the dial readings at 600 and 300 RPM respectively. The viscosity values were calculated using the relationship in Equation (6) as specified in the OFITE operational manual (1cp is equivalent to 10^{-3} Pas).

$$\eta = \text{KF}\frac{\theta}{\text{RPM}} \tag{6}$$

where η is the viscosity in cp, *K* is the machine constant of rotor – bob combination (R1B1) = 300, *F* is the spring factor = 1 for the R1B1 combination.

4. Development of rheological model

Based on the rheological results obtained using the viscometer, the bentonite mud (both the base case and the modified sample) exhibited shear thinning characteristic behaviour with yield point and this is non-linear. In order to fit a rheological model to predict this shear-thinning attribute, certain conditions must be satisfied (Vipulanandan & Mohammed, 2014):

$$\tau = \tau_0 \text{ when } \dot{\gamma} = 0 \tag{7}$$

$$\frac{d\tau}{d\dot{\gamma}} > 0 \tag{8}$$

$$\frac{d^2\tau}{d\dot{\gamma}^2} < 0 \tag{9}$$

$$\tau = \tau^{*} \text{ when } \dot{\gamma} = \infty \tag{10}$$

where τ is the shear stress, $\dot{\gamma}$ is the shear rate, τ_0 is the yield stress and τ^* is the maximum shear stress limit. Four rheological models were used in predicting the shear thinning attributes of the mud samples. The models were subjected to the conditions represented in Equations (7) to (10). The four models are: Herschel Bulkley, Sisko, Casson and Hyperbolic. Based on these conditions, it was observed that the Herschel Bulkley, Sisko and Casson models satisfies conditions (7) to (9) but not (10). In order words, they cannot predict the shear stress limit of the drilling fluid. The hyperbolic model on the other hand satisfies the conditions (7) to (10).

4.1. Herschel Bulkley rheological model

The Herschel Bulkley model relates shear stress to shear rate using three parameters which is represented mathematically in Equation (11).

$$\tau = \tau_{01} + K_1 \dot{\gamma}^{n} \tag{11}$$

where τ_{01}, τ, $\dot{\gamma}$, n, and K_1 represents the yield stress, shear stress, shear rate, flow constant and consistency factor respectively. Flow constant describes the shear thickening ($n > 1$) and shear thinning ($n < 1$) features of fluids. When $n = 1$ and $\tau_{01} = 0$, Equation (11) reduces to the Newtonian model.

4.2. Sisko rheological model

The Sisko model relates shear stress to shear rate using three parameters which is represented mathematically in Equation (12).

$$\tau = K_2 \dot{\gamma} + K_3 \dot{\gamma}^{m} \tag{12}$$

where K_2 is the coefficient of viscosity, K_3 is the consistency coefficient and m is the flow index of the fluid. $m < 1$ represents a pseudoplastic (shear thinning) fluid, $m > 1$ represents a dilatant (shear thickening) fluid and $m = 1$ correspond to a Newtonian fluid.

4.3. Casson rheological model

The Casson model relates shear stress to shear rate using two parameters which is represented mathematically in Equation (13).

$$\tau^{1/2} = \tau_{02}^{1/2} + K_4^{1/2} \dot{\gamma}^{1/2} \tag{13}$$

where K_4 and τ_{02} are the model constant and yield stress respectively.

4.4. Hyperbolic rheological model

A hyperbolic model similar to what was used by Vipulanandan and Mohammed (2014) was applied to investigate the rheological relationship of silica treated bentonite muds. The mathematical expression for the model is shown in Equation (14):

$$\tau = \tau_{03} + \frac{\dot{\gamma}}{\mathrm{A} + \mathrm{B}\dot{\gamma}} \tag{14}$$

where τ_{03} is the yield stress, A and B are the model parameters.

4.5. Accuracy of rheological model estimations

The accuracy and comparison of the various model predictions were quantified using the Root Mean Square Error (RMSE), residual plot analysis (A residual is the difference among the experimental *y*-value obtained from the scattered plot and the modelled y-value obtained from regression analysis) and the coefficient of determination (R^2) values obtained from the non-linear regression analysis of the constitutive models with the experimentally obtained rheological data. The RMSE and R^2 expressions are represented in Equations (15) and (16)

Table 2. Factor setting for response surface design

Variable	Notation	Unit	Level	
			Low	**High**
Bentonite content	*X*	wt.%	6.3	15
Silica nanoparticles	*Y*	wt.%	0	1.5

$$\mathrm{RMSE} = \sqrt{\frac{\sum_{i=1}^{n}(y_i - x_i)^2}{N}} \tag{15}$$

$$R^2 = \left(\frac{\sum_i (x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum_i (x_i - \bar{x})^2}\sqrt{\sum_i (y_i - \bar{y})^2}} \right)^2 \tag{16}$$

where y_i = experimental data obtained3 from rheological analysis, x_i = estimated data obtained from the fitted relationship, $\bar{y}$ = mean of the experimental data, $\bar{x}$ = mean of estimated data from fitted model and N = number of data points. The model parameters were obtained from nonlinear regression analysis of the various constitutive models with the experimental rheological data and are summarized in Tables 6a–6d.

4.6. Effect of bentonite content and silica nanoparticles on the rheological properties and maximum shear stress

The effect of bentonite content and amount of silica nanoparticles on the rheological properties and upper limit of shear stress of the bentonite mud was investigated using a response surface design. The response surface design was used with the experimental results to select factors (the bentonite content and amount of silica nanoparticles) that influence the maximum shear stress limit and rheological features (yield point, plastic viscosity, apparent viscosity) of the drilling mud significantly. The response surface design was carried out using Minitab 17 (Minitab Inc. USA). The range of factors used and the levels of the factors were based on the preliminary experimental results (Table 2). Analysis of Variance, which is also called ANOVA, was applied to estimate the suitability of using the response surface model after which optimization experimentations was used to obtain the suitable settings for the factors using the overlaid contour plot analysis (to determine the feasible region for the optimal values of the response variable) and the steepest method analysis (this analysis was based on the steepest ascent and descent path until the response neither increase or decrease). The response variables (rheological properties) were obtained based on Equations (3)–(5) while the maximum shear stress limit of the drilling mud was calculated based on Equation (14) using the hyperbolic model.

5. Results and discussion

5.1. Rheological properties of modified drilling mud

5.1.1. Response surface design

Optimization of factors like bentonite content (*X*) and amount of silica nanoparticles (*Y*) were examined. In this work, the effect of these parameters on the rheological properties (Plastic viscosity (PV), Apparent viscosity (AV) and Yield point (YP)) was determined through a screening procedure using a response surface design. Table 3 shows the outcome of the experiment carried out. The analysis of variance in Table 4 shows that the bentonite content, amount of silica nanoparticles and the interaction between them were significant factors (*p*-value of <0.05 was used as the limit point) which impacts the rheological properties (PV, YP and AV) of the silica nano-modified drilling mud. The linear

Table 3. The response surface design and the measured rheological properties (responses)

Run order	Bentonite content (wt.%)*	Silica nanoparticles (wt.%)*	Plastic viscosity (Pas)	Yield point (Pa)	Apparent viscosity (Pas)
	X	*Y*			
1	6.3	0.0	0.0034	2.1449	0.0055
2	6.3	0.5	0.0035	2.5535	0.0060
3	6.3	1.0	0.0036	2.5535	0.0061
4	6.3	1.5	0.0037	2.6046	0.0063
5	13.0	0.0	0.0120	22.982	0.0350
6	13.0	0.5	0.0130	25.535	0.0375
7	13.0	1.0	0.0130	28.089	0.0405
8	13.0	1.5	0.0130	31.663	0.0440
9	15.0	0.0	0.0170	62.816	0.0785
10	15.0	0.5	0.0230	68.945	0.0905
11	15.0	1.0	0.0280	66.391	0.0940
12	15.0	1.5	0.0300	67.923	0.0955

*Variables in un-coded levels.

Table 4. Effects and regression coefficients for plastic viscosity, yield point and apparent viscosity

Variable	Plastic viscosity			Yield point			Apparent viscosity		
	Estimated effect	Regression coefficient	*p*-value	Estimated effect	Regression coefficient	*p*-value	Estimated effect	Regression coefficient	*p*-value
Constant	–	0.00423	0.041	–	−0.430	0.009	–	0.00382	0.051
X	0.02095	0.01048	0.000	64.06	32.03	0.000	0.08365	0.04183	0.000
Y	0.00391	0.00196	0.049	4.275	2.137	0.025	0.00741	0.00370	0.004
X^2	0.01958	0.00979	0.002	69.83	34.92	0.000	0.08796	0.04398	0.000
XY	0.00492	0.00246	0.043	–	–	–	0.00718	0.00359	0.009

regression equations obtained from the regression analysis of the response surface design are listed below:

$$\mathrm{PV} = 0.0413 - 0.00918X - 0.00542Y + 0.000517X^2 + 0.000754XY \quad R^2 = 0.9655 \tag{17}$$

$$\mathrm{YP} = 128.32 - 31.94X + 2.85Y + 1.8453X^2 \quad R^2 = 0.991 \tag{18}$$

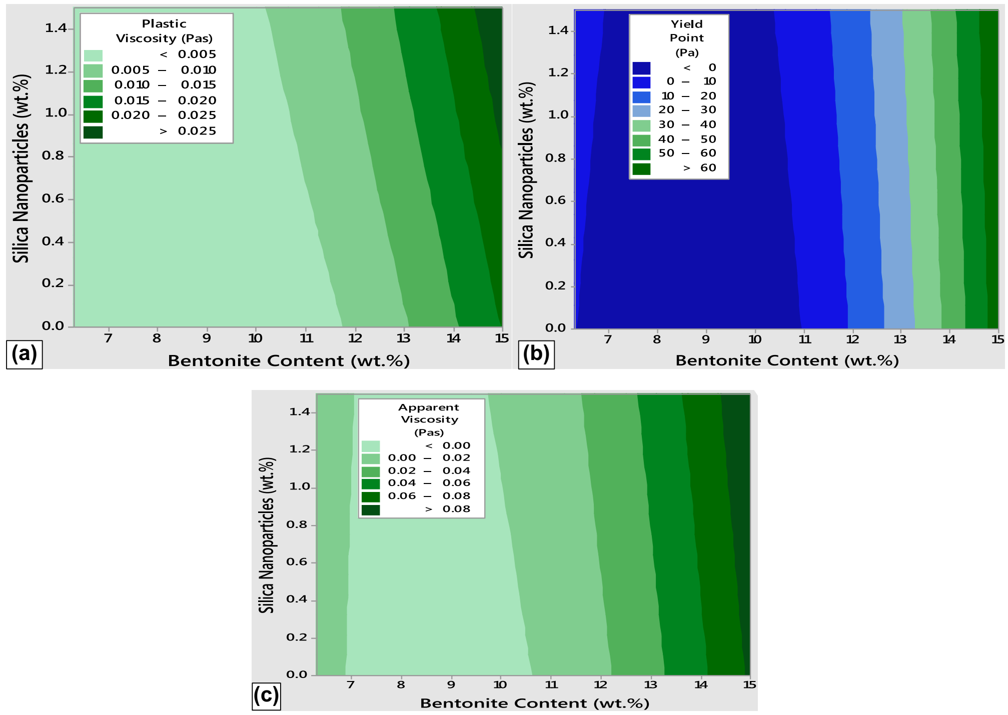
$$\mathrm{AV} = 0.1701 - 0.04072X - 0.00679Y + 0.002324X^2 + 0.001101XY \quad R^2 = 0.997 \tag{19}$$

From Equations (17) to (19), the bentonite content (*X*) and amount of silica nanoparticles (*Y*) have different impact on the response variables (PV, YP and AV). This indicates that in maximizing the rheological properties, the factors or terms in the regression equations with positive coefficients have to increase while those with negative coefficients must be decreased.

5.1.2. Overlaid contour plot analysis of rheological properties

Contour plots were produced from the regression Equations (17)–(19) obtained from the regression analysis using Minitab. Figure 1(a) shows the contour plot for plastic viscosity which showed that high plastic viscosity (>0.025 Pas) was attained at a combination of bentonite content 14.5–15 wt.% and silica nanoparticles greater than 0.9wt% while low plastic viscosity (< 0.005 Pas) was attained using a combination of bentonite content <10.2 wt.% and silica nanoparticles amounting between 0

Figure 1. Contour plots for (a) plastic viscosity, (b) yield point and (c) apparent viscosity.



and 1.5 wt.%. In the case of the yield point, a high yield point (>60 Pa) was gotten at a mixture of bentonite content ranging between 14.7 and 15 wt.% and silica nanoparticles >0 wt.% while a low yield point (< 20Pa) would require bentonite content < 12.8 wt.% and silica nanoparticles amounting between 0 and 1.5 wt.% (Figure 1(b)). For the apparent viscosity, a combination of bentonite content >14.4 wt.% and silica nanoparticles amounting to values >0 wt.% would give a high apparent viscosity (>0.08 Pas) (Figure 1(c)). In order to obtain a low apparent viscosity (< 0.02Pas), a combination of bentonite content <12.2 wt.% and silica nanoparticles ranging between 0 and 1.5 wt.% is required (Figure 1(c)). Other values of the rheological properties can be obtained between high and low using various combinations of the bentonite content and amount of silica nanoparticles. The plastic viscosity, yield point and apparent viscosity were overlaid using Equations (17), (18) and (19) and the contour plot to obtained the feasible region (white) having the preferred characteristics (Figure 2(a)). In overlaying, the bentonite content and amount of silica nanoparticles were the independent variables. The preferred values of all the rheological properties was acquired at any combination within the feasible (or optimized) region. The difference observed amongst the predicted and experimental values was found to be less than 4% based on random check.

5.1.3. Steepest analysis of rheological properties

In order to further optimize the rheological properties based on Equations (17) to (19), the method of steepest analysis was applied using the ASCENT macros in Minitab. The variable with the largest regression coefficient among Equations (17) to (19) was chosen as the base factor (bentonite content). Table 5 shows the result of the steepest analysis and this indicates that decrease in the rheological properties was observed through the 7th step after which steps beyond this point resulted in an increase in the rheological properties. Therefore, factors at the 7th step were selected as the optimal variables which minimizes the rheological properties. The optimal solution can also be obtained in feasible region (white) of the overlaid contour plots of Figure 2(a).

5.1.4. Effect of bentonite content and silica nanoparticles on rheological properties

The introduction of silica nanoparticles into the water based bentonite mud lead to an increase in the values of the rheological properties than the ordinary bentonite mud without the silica nanoparticles (Figure 2(b)–(d)]. The improved rheological properties can be explained in terms of the

Figure 2. (a) Overlaid contour plot for plastic viscosity, yield point and apparent viscosity, (b) effect of silica nanoparticles on plastic viscosity, (c) effect of silica nanoparticles on yield point and (d) effect of silica nanoparticles on apparent viscosity.

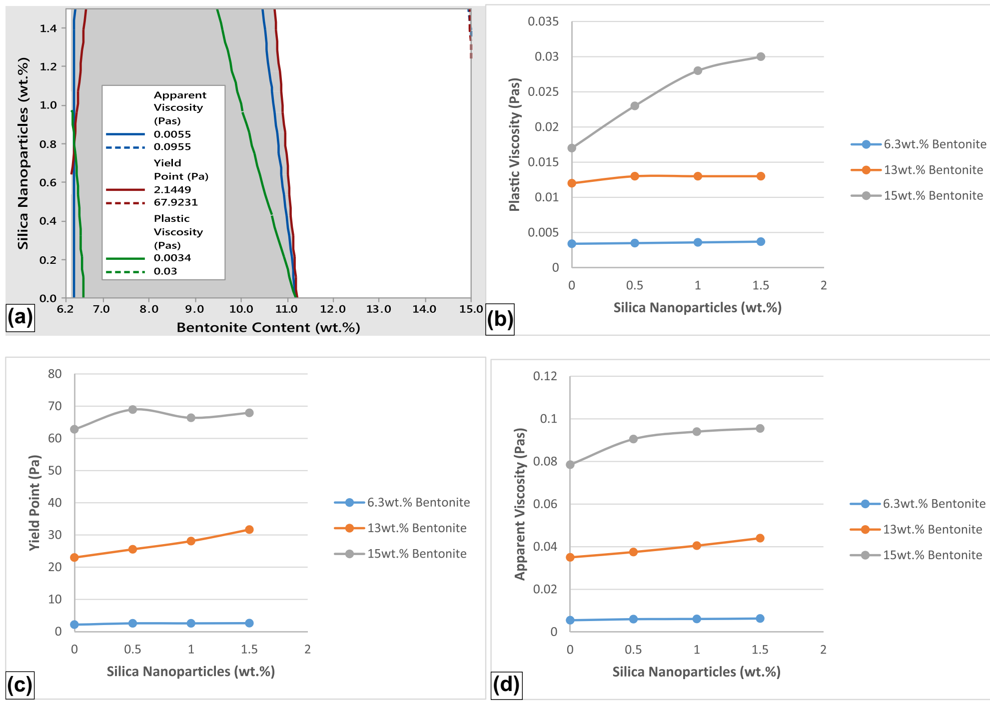


Table 5. Points along the path for steepest ascent and descent and observed plastic viscosity, yield point and apparent viscosity of sample at the points

Run No.		Bentonite content (wt.%)	Silica nanoparticles (wt.%)	Plastic viscosity (Pas)	Yield point (Pa)	Apparent viscosity (Pas)
	Base	10.65	0.750	0.00413	−0.40496	0.00373
1	Base - 6Δ_i	36.75	−0.390	0.39348	1,445.60	1.79922
2	Base - 5Δ_i	32.40	−0.200	0.28279	1,030.01	1.28464
3	Base - 4Δ_i	28.05	−0.010	0.19042	684.261	0.85619
4	Base - 3Δ_i	23.70	0.180	0.11637	408.341	0.51388
5	Base - 2Δ_i	19.35	0.370	0.06064	202.257	0.25769
6	Base - Δ_i	15.00	0.560	0.02322	66.0085	0.08765
	Δ_i	−4.350	0.190			
7	Base + Δ_i	6.300	0.940	0.00336	3.01695	0.00594
8	Base + 2Δ_i	1.950	1.130	0.02090	76.2742	0.09429
9	Base + 3Δ_i	−2.400	1.320	0.05676	219.366	0.26876
10	Base + 4Δ_i	−6.750	1.510	0.11095	432.295	0.52937
11	Base + 5Δ_i	−11.10	1.700	0.18346	715.058	0.87611
12	Base + 6Δ_i	−62.50	7.440	0.27428	1,067.65	1.30899

dispersion ability of the silica nanoparticles to be well distributed and interact more efficiently on the bentonite clays. Previous characterization of bentonite clay samples has shown that silicon dioxide (or silica) make a large proportion of the chemical composition of bentonite clays (James, Mesubi, Adekola, Odebunmi, & Adekeye, 2008; Karnland, 2010; Lim, Gomes, & Kadir, 2013; Omole, Adeleye, Falode, Malomo, & Oyedeji, 2013). It could be suggested that that the interaction between the silica nanoparticles and the constituent silica compounds in the bentonite clays may explain the increased rheological properties of the silica modified drilling mud. Nanoparticles have high surface areas per volume and this will allow for communication of the nanoparticles with the bentonite matrix and surrounding water-based drilling fluid. The surface area of nanoparticles tends to act as active sites for attachment with functional groups and can impact on chain entanglement and thus produce a variation in the properties of the matrix. Thus, the nanoparticles and base fluid may be linked or bonded together directly or through certain intermediate chemical linkages to improve the rheological attributes of water based drilling mud. This could also be explained by consideration of synergy of homo-coagulation of between silica nanoparticles and hetero-coagulation of silica nanoparticles with bentonite particles in the suspension (Ismail, Aftab, Ibupoto, & Zolkifile, 2016; Jung et al., 2011). These observations are also corroborated by the regression analysis carried out in Equations (17)–(19) which shows that the interaction (denoted *XY*) between bentonite content (*X*) and amount of silica nanoparticles (*Y*) likewise the interaction between clay particles (denoted X^2) have to be increased in order to have increased plastic viscosity and apparent viscosity. The yield point on the order tend to increase due to accumulation of solids resulting from the increased amount of nanoparticles (*Y*) and interaction between bentonite particles (X^2).

5.2. Rheological models for nano-modified drilling mud

Flow attributes and characteristics of the modified bentonite mud was modelled based on the rheological relationship using a hyperbolic model and this was compared to the Herschel Bulkley, Casson and Sisko models as shown in Figures 3–5.

5.2.1. Herschel Bulkley rheological model

The rheological relationship of the mud containing and without silica nanoparticles was modelled with Herschel Bulkley rheological relationship (Equation (11)) with a shear rate reaching 1,021.8 s^{-1}. The coefficient of determination, R^2, values for the base case (without silica nanoparticles) was 0.999, 0.998, 0.998 for 6.3, 13 and 15 wt.% bentonite content respectively (Table 6a). The RMSE for the base case samples varied from 0.22Pa for 6.3 wt.% bentonite content to 2.41 and 6.96 Pa for 13 and 15 wt.% bentonite content respectively. The R^2 and RMSE values of nano-modified drilling mud containing up to 1.5 wt.% silica nanoparticles are summarized in Table 6a. The model parameters, yield stress (τ_{01}) and consistency index (K_1), show an increasing trend with increasing amount of bentonite and silica nanoparticles respectively as shown in Tables 6a. The flow index, *n*, decreases with increasing quantity of bentonite.

Figure 3. Predicted and measured shear stress-shear rate data for 6.3 wt.% bentonite mud containing different amount of silica nanoparticles (a) 0 wt.%, (b) 0.5 wt.%, (c) 1 wt.% and (d) 1.5 wt.%.

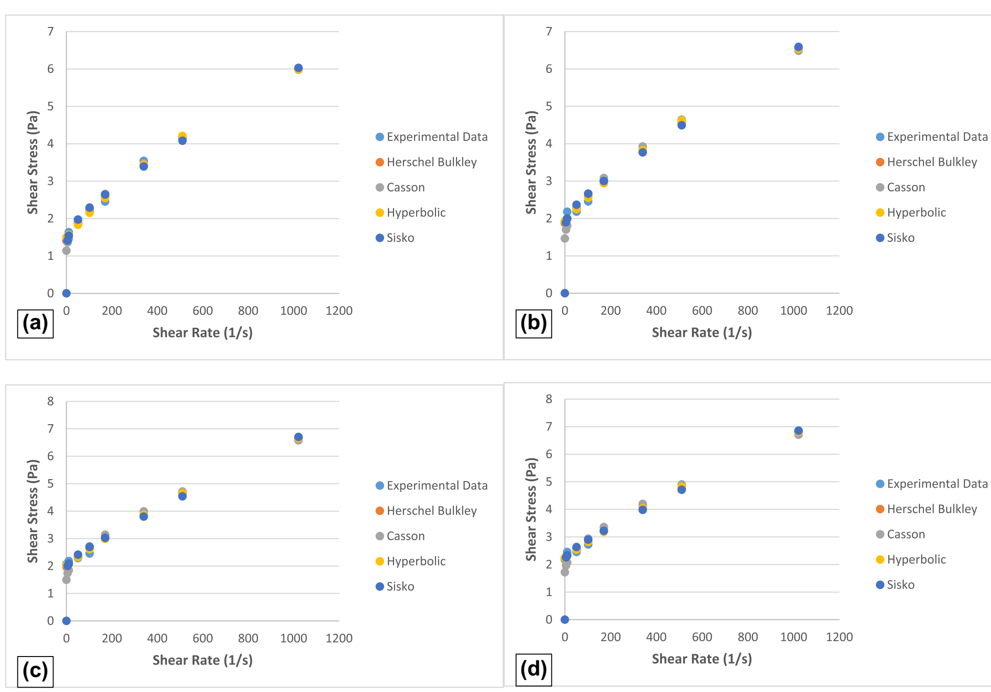


Figure 4. Predicted and measured shear stress-shear rate data for 13 wt.% bentonite mud containing different amount of silica nanoparticles (a) 0 wt.%, (b) 0.5 wt.%, (c) 1 wt.% and (d) 1.5 wt.%.

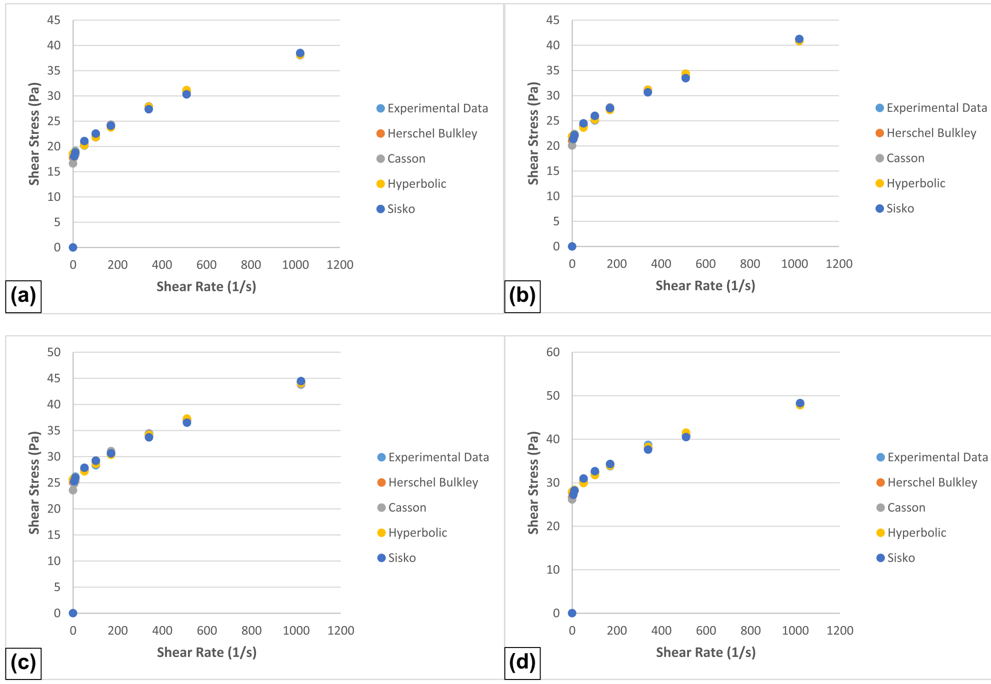
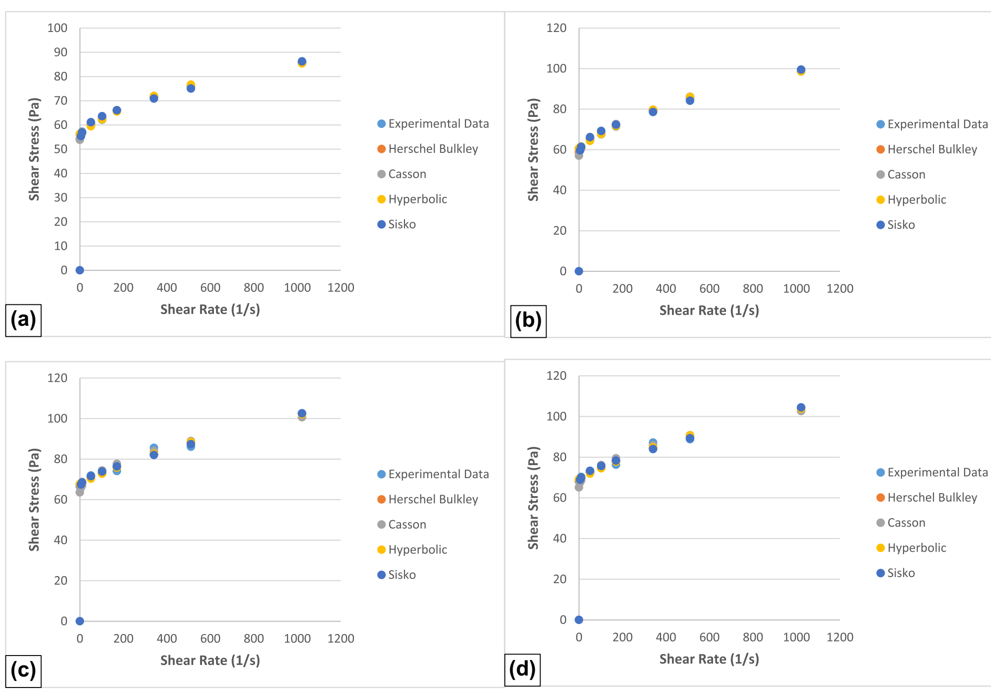


Figure 5. Predicted and measured shear stress-shear rate data for 15 wt.% bentonite mud containing different amount of silica nanoparticles (a) 0 wt.%, (b) 0.5 wt.%, (c) 1 wt.% and (d) 1.5 wt.%.



5.2.2. Sisko rheological model

Shear thinning behaviour of the drilling mud was modelled using Sisko model. The Sisko model (Equation 12) is a three parameter model (K_2, K_3 and m). The R^2 values for the base case (without silica nanoparticles) was 0.964, 0.828, 0.688 for 6.3, 13 and 15 wt.% bentonite content respectively (Table 6d). The RMSE for the base case samples varied from 0.31 Pa for 6.3 wt.% bentonite content to 4.93 and 14.09 Pa for 13 and 15 wt.% bentonite content respectively. The R^2 and RMSE values of nano-modified drilling mud containing up to 1.5 wt.% silica nanoparticles are summarized in

Table 6d. The model parameters, (K_1) and (K_2), show an increasing trend with increasing amount of bentonite and silica nanoparticles respectively as shown in Table 6d. The index, m, decreases with increasing amount of bentonite content and silica nanoparticles (Table 6d).

5.2.3. Casson rheological model

The Casson model relates shear stress to shear rate using 2 parameters which is represented mathematically in Equation (13). The values for the coefficient of determination, R^2, for the base case (without silica nanoparticles) was 0.996, 0.994, 0.996 for 6.3, 13 and 15 wt.%bentonite content respectively (Tables 6a–6d). The RMSE for the base case samples varied from 0.26 Pa for 6.3 wt.% bentonite content to 3.47 and 6.97 Pa for 13 and 15 wt.% bentonite content respectively. The R^2 and RMSE values of nano-modified drilling mud containing up to 1.5 wt.% silica nanoparticles are summarized in Table 6c. The model parameters, (τ_{02}) and (K_4), show an increasing trend with increasing amount of bentonite and silica nanoparticles respectively as shown in Table 6c.

5.2.4. Hyperbolic rheological model

A hyperbolic model similar to what was used by Vipulanandan and Mohammed (2014) was used to investigate the rheological relationship of silica nanoparticle treated drilling muds. The mathematical expression for the model is shown in Equation (14). The hyperbolic model consist of 3 parameters (τ_{03}, A and B). The R^2 (coefficient of determination) values for the base case (without silica nanoparticles) was 0.999, 0.999, 0.999 for 6.3, 13 and 15 wt.% bentonite content respectively (Table 6b). RMSE for the base case samples varied from 0.19 Pa for 6.3 wt.% bentonite content to 1.00 and 4.30 Pa for 13 and 15 wt.% bentonite content respectively. The R^2 and RMSE values of nano-modified drilling mud containing up to 1.5 wt.% silica nanoparticles are summarized in Table 6b. The model parameter (τ_{03}) increased with bentonite content and amount of silica nanoparticles while the other parameters, A and B, show a decreasing trend with increasing amount of bentonite content and silica nanoparticles (Table 6b).

5.2.5. Comparison between rheological models

The various rheological models applied in this work were statistically evaluated using R^2 value, RMSE and residual plot analysis. The coefficient of determination showed that the Herschel Bulkley model accounted for 99.1% to 99.9% of the variance between the experimental data points for the rheological relationship and the fitted regression model as shown in Table 6a. The Sisko model accounted for between 67.4 and 96.4% of the variance while the Casson model accounted for 98.8 to 99.6% of the variance. The new hyperbolic model accounted for 99.9% of the variance. From the coefficient of determination analysis, the Sisko model does not account for much of the variance between the experimental data and the fitted model. Based on the use of the R^2 values alone, it could be

Table 6a. Herschel Bulkley model parameters for silica nano-modified bentonite drilling mud

Bentonite (wt.%)	Silica nanoparticles (wt.%)	Herschel Bulkley model (Equation 11)				
		τ_{01} (Pa)	K_1 [(Pa)s^n]	n	RMSE (Pa)	R^2
6.3	0.0	1.399	0.02605	0.747	0.221	0.999
	0.5	1.869	0.01787	0.803	0.279	0.998
	1.0	1.959	0.01481	0.834	0.266	0.998
	1.5	2.217	0.01277	0.853	0.253	0.998
13	0.0	17.76	0.20683	0.664	2.408	0.998
	0.5	21.03	0.24105	0.638	2.701	0.998
	1.0	25.04	0.15423	0.696	2.730	0.999
	1.5	26.63	0.38303	0.581	3.740	0.997
15	0.0	54.29	0.66851	0.556	6.966	0.998
	0.5	58.83	0.56228	0.616	9.657	0.998
	1.0	67.36	0.17568	0.764	24.67	0.991
	1.5	68.64	0.22317	0.731	20.53	0.994

Table 6b. Hyperbolic model parameters for silica nano-modified bentonite drilling mud

Bentonite (wt.%)	Silica nanoparticles (wt.%)	Hyperbolic model (Equation 14)				
		τ_{03} (Pa)	A [(Pa)s]$^{-1}$	B [Pa]$^{-1}$	RMSE (Pa)	R^2
6.3	0.0	1.501	39.02	0.020	0.193	0.999
	0.5	1.935	41.49	0.016	0.203	0.999
	1.0	2.025	43.78	0.013	0.178	0.999
	1.5	2.244	45.06	0.013	0.199	0.999
13	0.0	18.54	7.533	0.006	1.002	0.999
	0.5	21.88	7.349	0.007	1.419	0.999
	1.0	25.65	8.467	0.007	1.621	0.999
	1.5	27.87	6.169	0.006	3.349	0.999
15	0.0	56.28	3.932	0.005	4.301	0.999
	0.5	60.72	3.494	0.004	5.812	0.999
	1.0	67.82	5.080	0.003	24.11	0.999
	1.5	69.20	4.692	0.003	19.56	0.999

Table 6c. Casson model parameters for silica nano-modified bentonite drilling mud

Bentonite (wt.%)	Silica nanoparticles (wt.%)	Casson model (Equation 13)			
		τ_{02} (Pa)	K_4 [(Pa)s^{-1}]	RMSE (Pa)	R^2
6.3	0.0	1.140	0.0020	0.264	0.996
	0.5	1.463	0.0015	0.445	0.991
	1.0	1.498	0.0015	0.511	0.988
	1.5	1.715	0.0015	0.572	0.988
13	0.0	16.62	0.0041	3.467	0.994
	0.5	20.06	0.0036	3.431	0.995
	1.0	23.54	0.0031	5.133	0.991
	1.5	26.15	0.0031	3.725	0.996
15	0.0	53.83	0.0036	6.971	0.996
	0.5	57.05	0.0056	11.43	0.995
	1.0	63.58	0.0041	30.01	0.982
	1.5	65.17	0.0041	25.94	0.986

Table 6d. Sisko model parameters for silica nano-modified bentonite drilling mud

Bentonite (wt.%)	Silica nanoparticles (wt.%)	Sisko model (Equation 12)				
		K_2 [(Pa)s^{-1}]	K_3 [(Pa)s^m]	m	RMSE (Pa)	R^2
6.3	0.0	0.0036	1.165	0.110	0.310	0.964
	0.5	0.0041	1.697	0.062	0.381	0.941
	1.0	0.0041	1.835	0.047	0.371	0.931
	1.5	0.0041	2.109	0.036	0.341	0.927
13	0.0	0.0143	16.48	0.053	4.930	0.828
	0.5	0.0133	19.69	0.048	5.245	0.792
	1.0	0.0143	23.92	0.032	5.075	0.742
	1.5	0.0133	25.12	0.048	6.654	0.755
15	0.0	0.0189	52.04	0.037	14.10	0.688
	0.5	0.0266	56.02	0.037	17.85	0.722
	1.0	0.0281	65.42	0.018	25.31	0.676
	1.5	0.0276	66.60	0.019	22.79	0.674

concluded that the hyperbolic model gave a better correlation of how close the experimental shear stress-shear rate are to the fitted model. The coefficient of determination alone cannot state the suitability of a fitted model and whether the coefficient estimations and predictions are partial. Hence the need to consider the residual plots and the RMSE of the models. The residual plot analysis of the Hyperbolic, Casson and Herschel Bulkley models for 6.3 wt.% bentonite containing 0 to 1.5 wt.% silica nanoparticles is shown in Figure 6. The residual plots for the Herschel Bulkley model and the Hyperbolic model indicated that there is a random variation in the plot of the residuals with the fitted data points. There does not seem to be any strong pattern among the points in this plot as they tend to cluster towards the middle of the plot, hence the two models gave a good fit to the experimental data. For the Casson model, there was a systematic pattern of deviation in the plot of the residuals and the fits. They are systematically increasing and decreasing along the horizontal axis. The RMSE values for the Herschel Bulkley and Hyperbolic models showed that values for the Hyperbolic model is less than that of the Herschel Bulkley model (Tables 6a–6d). Therefore, it can be concluded that the Hyperbolic model predicts the experimental rheological data satisfactorily.

5.3. Maximum or upper shear stress limit for modified drilling mud

The Hyperbolic model based on Equation (14) has a maximum or cap on the shear stress the bentonite mud can tolerate at extreme shear rates. The shear stress limit was calculated using Equation (20).

$$\dot{\gamma} = \infty \text{ therefore } \tau^* = \frac{1}{B} + \tau_{03} \tag{20}$$

The effect of bentonite content (X) and silica nanoparticles on the maximum shear stress limit, τ^*, was determined through a screening process using a response surface design. Table 8a shows the result of the analysis carried out. The analysis of variance in Table 8b points to the fact that the bentonite content, amount of silica nanoparticles likewise the interaction between them were significant factors (p-value of <0.05 was used as the limit for significance differences) which affects the maximum shear stress limit of the silica nano-modified bentonite mud. The linear regression equation obtained from the regression analysis of the response surface design is listed below:

$$\tau^* = 286.3 - 65.33X - 18.9Y + 3.613X^2 + 3.23XY \quad R^2 = 0.9816 \tag{21}$$

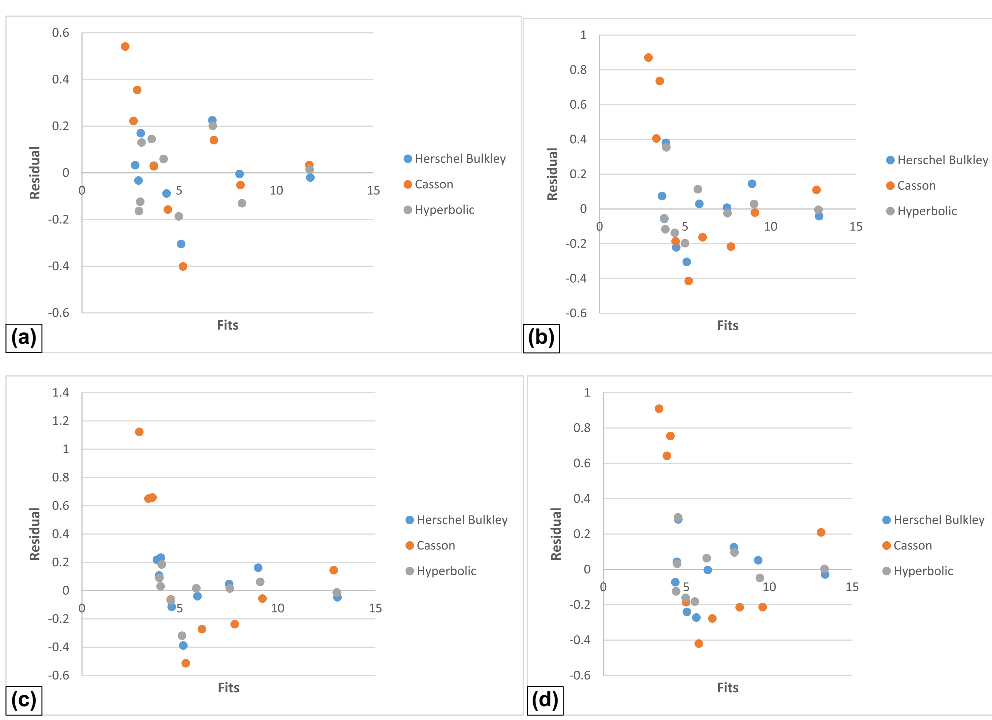


Figure 6. Residual plot for 6.3 wt.% bentonite mud containing different amount of silica nanoparticles (a) 0 wt.%, (b) 0.5 wt.%, (c) 1 wt.% and (d) 1.5 wt.%.

Table 7. Points along the path for steepest ascent and descent and observed maximum shear stress tolerance of sample at the points

Run no.		Bentonite content (wt.%)	Silica nanoparticles (wt.%)	Shear stress limit (Pa)
	Base	10.65	0.750	12.01
1	Base - 6Δ_i	36.75	−0.390	2,725
2	Base - 5Δ_i	32.40	−0.200	1,945
3	Base - 4Δ_i	28.05	−0.010	1,296
4	Base - 3Δ_i	23.70	0.180	777.8
5	Base - 2Δ_i	19.35	0.370	391.2
6	Base - Δ_i	15.00	0.560	135.9
	Δ_i	−4.350	0.190	
7	Base + Δ_i	6.300	0.940	19.51
8	Base + 2Δ_i	1.950	1.130	158.4
9	Base + 3Δ_i	−2.400	1.320	428.6
10	Base + 4Δ_i	−6.750	1.510	830.4
11	Base + 5Δ_i	−11.10	1.700	12.01
12	Base + 6Δ_i	−62.50	7.440	2,726

Table 8a. The response surface design and the estimated maximum shear stress limit from Equation (20)

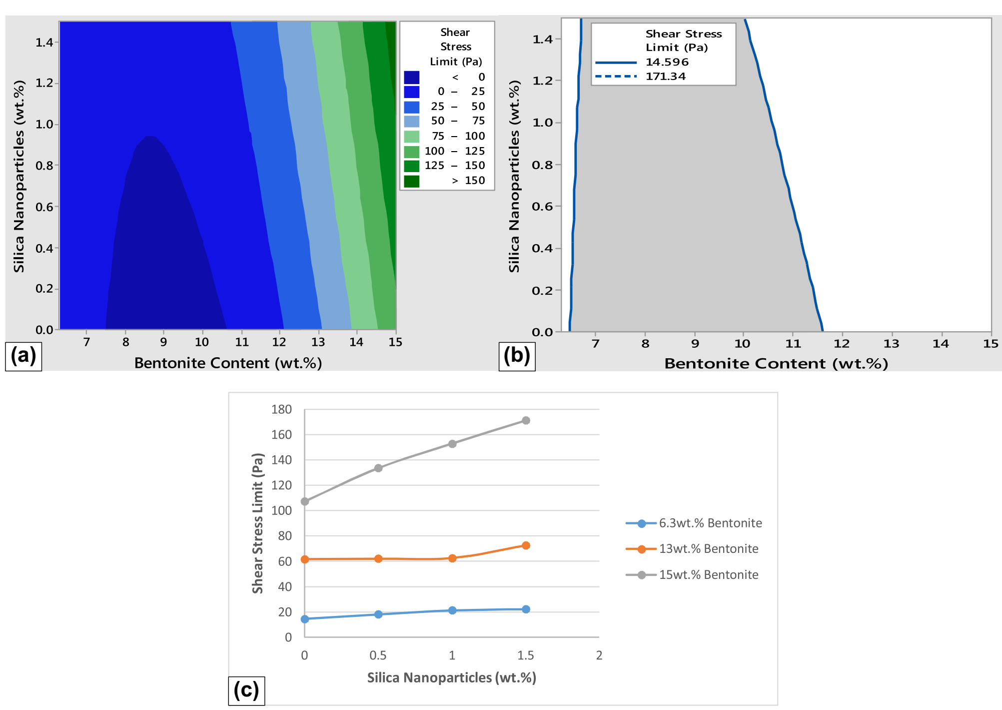
Run order	Bentonite content (wt.%)	Silica nanoparticles (wt.%)	Shear stress limit (Pa)
	X	*Y*	
1	6.3	0.0	14.60
2	6.3	0.5	18.25
3	6.3	1.0	21.37
4	6.3	1.5	22.27
5	13.0	0.0	61.74
6	13.0	0.5	62.20
7	13.0	1.0	62.66
8	13.0	1.5	72.62
9	15.0	0.0	107.4
10	15.0	0.5	133.7
11	15.0	1.0	152.9
12	15.0	1.5	171.3

Table 8b. Effects and regression coefficients for maximum shear stress tolerance

Variable	Shear stress limit		
	Estimated effect	Regression coefficient	*p*-value
Constant	–	11.85	0.014
X	122.2	61.09	0.000
Y	23.25	11.62	0.016
X^2	136.7	68.37	0.000
XY	21.09	10.54	0.041

From on Equation (21), the bentonite content (*X*) and amount of silica nanoparticles (*Y*) have different impact on the maximum shear stress limit. This shows that in maximizing the shear stress limit, the factors or terms in the equation with positive coefficients have to increase while those with negative coefficients must be decreased. Contour plot produced from the regression equation showed that high shear stress limit (>150 Pa) was found at a combination of bentonite content 14.2–15 wt.% and silica nanoparticles greater than 0.4 wt.% (Figure 7(a)) while low maximum shear stress limit (<25.5 Pa) was gotten using a combination of bentonite content <12 wt.% and silica nanoparticles amounting between 0 and 1.5 wt.% (Figure 7(a)). For the overlaid contour plot analysis, the bentonite content and amount of silica nanoparticles were the independent variables. The preferred values of the maximum shear stress limit could be acquired at any combination within the feasible (white) region (Figure 7(b)). Further optimization of the shear stress limit based on the method of steepest analysis was applied using the ASCENT macros in Minitab. The variable with the largest regression coefficient among Equation (21) was chosen as the base factor (bentonite content). Table 7 shows the outcome of the steepest analysis and this indicates that a decrease in the maximum shear stress limit was achieved at the 7th step after which steps after this point resulted in high values for the maximum shear stress limit. Therefore, factors at the 7th step were chosen as the optimal variables which minimizes the maximum shear stress limit. The optimal solution can also be obtained in feasible region (white) of the overlaid contour plot of Figure 7(b). The maximum shear stress limit of the drilling mud was observed to increase with increasing bentonite content and amount of silica nanoparticles (Figure 7(c)). Equation (21) shows the regression model of the relationship between the bentonite content and silica nanoparticles. This demonstrates that the interaction (denoted *XY*) between bentonite content (*X*) and amount of silica nanoparticles (*Y*) likewise the interaction between clay particles (denoted R^2) have to be increased in order to have increased maximum shear stress limit. Theoretically, the synergy between the interaction between bentonite clay particles and that between bentonite and silica nanoparticles tend to explain the effect on the maximum shear stress limit of the nano-modified drilling mud. In order words, the shear stress limit of the nano-modified mud is enhanced by the interaction (bonding) of the silica nanoparticles within the clay matrix. The large surface area of the nanoparticles provides this bonding sites with the clay particles.

Figure 7. (a) Contour plot for shear stress limit, (b) overlaid contour plot for shear stress limit and (c) effect of silica nanoparticles and bentonite content on maximum shear stress limit.



6. Conclusion

In this work, the rheological characteristics and maximum or upper shear stress limit of bentonite mud treated with silica nanoparticles was studied. The bentonite mud was modified with up to 1.5% silica nanoparticles by weight. Based on this investigation and rheological modelling, the following conclusions can be drawn:

(1) The addition of silica nanoparticles up to 1.5 wt.% modified the rheological properties and shear stress limit of the bentonite mud. The changes observed in the properties are as a result of the bentonite content and amount of silica nanoparticles in the mud.

(2) The influence of bentonite content and amount of silica nanoparticles on the rheological properties was quantified using a response surface design. An overlaid contour plot and steep method was used to identify the optimal combination of bentonite and silica nanoparticles.

(3) The maximum shear stress limit was predicted using a hyperbolic model while the traditional oil industry models (Herschel Bulkley, Casson, Sisko etc.) can only predict an infinite shear stress limit.

(4) The hyperbolic model was a good fit for the shear stress-shear rate relationship. This was concluded based on the residual plot analysis, root mean square error and coefficient of determination.

(5) The hyperbolic relationship estimated the upper shear stress limit produced by 6.3, 13 and 15 wt.% mud as 14.59, 61.74 and 107.4 Pa respectively. The upper shear stress limit of a 1.5 wt.% silica nanoparticle treated 6.3, 13 and 15 wt.% bentonite mud were 22.27, 72.62 and 171.3 Pa respectively.

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