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Synthesis of Nanosilica and its application as Water Based Mud Rheology Enhancer

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Abstract. This study synthesized nanosilica from sodium silicate solution using precipitation method for 3:1 ammonia/ ethanol ratio, and examines the effectiveness of the nanosilica in formulating high-performance water based mud system. Sodium silicate solution (SSS) is a low-cost precursor than tetraethyl orthosilicate. Three (3) solutions of sodium silicate with concentrations 0.5 N, 1 N and 1.5 N were used to precipitate nanosilica. The use of sodium silicate solution in alkaline medium produces less agglomerated nanosilica. The results gave Similarly, three (3) different samples of silica nanoparticles (Sample A, Sample B, and Sample C) with each concentration respectively. The silica nanoparticles from the Sample A had the smallest in size and quantity, and also the most dispersed. For Sample B, the nanosilica produced was apparently bigger than that of sample A in size and quantity produced. Sample C was produced using two procedures: Sample C1 (with ultra Sonicator) and Sample C2. (ultra Sonicator). Samples C1 and C2 produced the biggest size and quantity of nanosilica with less the dispersion. But, there were aggregation problems, due to the higher concentration of sodium silicate solution. There was no much difference between samples C1 and C2; just that with the former, the nanosilica was better dispersed and the latter was little more aggregated. The precipitated nanosilica, Sample B with 1 N concentration of SSS gave the optimum size and quantity, and was used to formulate the examined high-performance water based mud system. The silica nanoparticles had a positive effect on the drilling fluid systems flow properties. This due to their ultrafine size and high surface area. Also, nanosilica production is cheap and feasible, so it could easily replace other expensive additives.

Keywords: Synthesis; Nanosilica; Oil and gas; Water based mud; Flow properties; Sodium silicate solution

1. Introduction

The success and cost of an oil and gas well depends largely on the characteristics of the drilling fluid. Although the drilling fluid cost itself is relatively low, choosing the right fluid and maintaining the correct drilling characteristics can have a significant impact on the total cost of a well [1]. Therefore, choosing the right drilling fluid and managing its properties on a daily basis is key to a successful drilling operation. Often, many requirements or functions are imposed on the drilling fluid [2, 3]. Over the years, a large number of drilling fluid formulations have been developed to provide different subsurface scenarios. Choosing the right drilling fluid system for the expected drilling conditions minimizes cost of well and reduces the possible risk of instability.

Functional requirements for drilling fluid systems have changed from just the removal of drill-cuttings to many, which include pressure control and maintaining wellbore stability [4]. At the same time, the mud system must not adversely affect productivity or harm personnel, drilling equipment or the environment. Most drilling fluids are complex mixtures that vary in flow properties with varying temperature and shear speed. As the drilling fluid circulates through the drill pipe to the borehole, it constantly changes, for example, the flow inside the drill-pipes is often associated with turbulence at the bit nozzle, and the annulus flow behaviour related to laminar flow at regular shear rate during circulation. In addition, during the circulation of the drilling fluid, a constant temperature change occurs, and the constant composition changes as solutes and fluids from the subsurface formation are introduced into the drilling fluid during drilling operation.

Literature have shown the use of additives in improving drilling mud systems rheological properties, and the effect of drilling technology developments on drilling mud additives [5, 6]. The emphasis in recent years in the drilling industry has been on restraining the increase in cost of drilling a well by designing mud system optimizes drilling operation. It is worth noting that the performance of a drilling fluid system notwithstanding the type is dependent on the additives. There is an emergent demand to develop additives with precise features not often provided by a single constituent [7]. According to Vryzas and Kelessidis [8], nanoparticles are making a significant contribution to technological progress in various fields and are the most promising material/ particles for the development of intelligent drilling mud systems with special properties and features that meet the complex subsurface requirements. The inclusion of inert inorganic nanoparticles can increase the abrasion resistance and antibacterial properties of the water based drilling mud system [9].

Silicon dioxide nanoparticles are the basis of most research due to their thermal stability, low toxicity and ability to work with a number of molecules and polymers. Nanosilica as it is often called, have played a prominent role in scientific research due to their ease of manufacture and widespread use in various industrial applications [10]. The quality of some of these products is highly dependent on the size distribution of these particles. It is present in almost all minerals on the Earth's surface. For commercial purposes, pure silicon is usually obtained from quartz rock and sodium silicates [11]. Therefore, the use of nanosilica as an additive in drilling fluid will not affect performance as silicon is naturally found in the subsurface environment.

Water based mud system naturally is environmentally-friendly due to its compositions, but their flow properties exhibits instability characteristics when open to high temperature [12]. There is numerous literature on stabilization of water based drilling mud to optimize the flow properties during drilling operation. Most of these studies attempted to incorporate different nanoparticles into water based mud systems for rheological properties optimization. This study synthesized nanosilica from sodium silicate solution, and investigates the effect of nanosilica spontaneous gelation tendency in water based drilling fluid system rheology. To successfully complete a drilling operation, a mud system whose flow properties remain stable during drilling operation is required.

2 Methods and Materials

2.1 Synthesis of Nanosilica

Top-down and bottom-up methods are two types of techniques adopted in nanofabrication. The bottom-up approach is superior to the top-down approach because the former has a better chance of having less defects, a more uniform chemical composition, and better control over short and long distances. In the bottom-up synthesis method, nanostructures are synthesized on the substrate by sticking atoms on top of each other, which leads to the formation of crystal planes, and the crystal planes lay on top of each other to synthesize nanostructures. Therefore, the bottom-up approach can be considered as a synthetic approach that is added to the substrate to create nanostructures. There are different ways for synthesizing nanosilica like colloidal method and sol-gel method. Because of the need to have the nanosilica in its powder form for mud system additive, the present study adopted the "precipitation method", which is also known as "colloidal method".

In this study, the Sodium Silicate Solution (SSS) was prepared with Sodium meta Silicate hydrate at different concentrations: 0.5N, 1N, and 1.5N. Diluted SSS and mixture of ammonia and ethanol was used to synthesize nanosilica in this study. The experiment was carried out in an alkaline medium because the silica nanoparticles are better dispersed and reduced in an alkaline medium to avoid agglomeration issues. To observe the effect of the process on the size, distribution and production volume of nanosilica, each SSS was diluted with distilled water and treated with a 3:1 mixture of ammonia and ethanol. Zulfigar et al. [13] in their study highlighted that a 3:1 Ammonia/Ethanol ratio will aid production of nanosilica.

The SEM was used to observe the particle morphology of the produced nanosilica.

2.2 Procedures adopted in Production of Nanosilica

Sodium Silicate Solution (SSS) Preparation

1. 3.55 g, 7.1 g, and 10.65 g of sodium silicate were weighed.
2. 25 ml of distilled water was measured for each preparation.
3. Sodium silicate was mixed with the distilled water while stirring, and taking the ultrasonic bath to temperature of 50 ^oC at 40 Hz frequency for 30min.

This steps were repeated in preparing different sample concentration of 0.5N, 1N, and 1.5N (see Figure 1). Table 1 shows the details of the sodium silicate solution used to precipitate the different samples of the nanosilica in this study.

Table 1. Samples of Sodium Silicate Solution

Nanosilica Samples	Sodium Silicate (g)	Distilled Water (ml)	Concentration (N)
A	3.55	25	0.5
B	7.10	25	1.0
C	10.65	25	1.5

Procedure "A": With Ultra Sonication Bath

1. Prepare a mixture with ammonia and ethanol in 3:1 ratio, that is, 90 ml of Ammonia and 30ml of Ethanol. Ultrasonic bath was used for 10min during the mixture.
2. In the precursor medium of silica, add 2 ml of SSS in 7 ml of distilled water; and then, add the mixture drop-wise to Ammonia/ Ethanol mixture (add while sonicating).
3. Use ultrasonic bath for 1-hour aging.
4. After, centrifuge at 4000 RPM and wash with distilled water to obtain the silica nanoparticles.

Figure 1. Sodium Silicate Solution at different concentrations: 0.5 N, 1 N, and 1.5 N

Procedure "B": Without Ultra Sonication Bath

1. Prepare a mixture with ammonia and ethanol in 3:1 ratio, that is, 90 ml of Ammonia and 30ml of Ethanol.
2. In the precursor medium of silica, add 2 ml of SSS in 7 ml of distilled water; and then, add the mixture drop-wise to Ammonia/ Ethanol mixture.
3. After 1 h of heating, centrifugation was performed at 4000 rpm and the solutes washed with distilled water to obtain silicon nanosilica.

2.3 Water Based Drilling Fluid Preparation

The materials/ additives used in the formulation of the water based mud sample are tabulated in Table 2 with their mixing order. The flow properties were measured in accordance with API standards. The mud system samples were mixed with Hamilton Beach mixer in Covenant University Drilling Fluids Laboratory, and the individual flow properties examined accordingly.

Table 2. Laboratory formulation for equivalent WBM field barrel

Product Name	Mixing time (mins)	S.G	Product conc. for 1 lab bbl (350mls)
			lbs
Base Fluid	1	1	330
Viscosifier	2	1.5	1.40
Fluid loss Additive	2		-
Alkalinity	3	2.5	0.15
Salt	-	3.31	10.61
Other	2	2.13	0.25
Others	3	1.07	0.25
Barite	8	3.9	69.0

3. Results and Discussion

The choice of nanosilica in this study is related to its excellent physicochemical properties which has a positive effect in the water based mud flow properties.

3.1 Synthesized Nanosilica

According to the investigation on the effect of Ethanol volume on the nanoparticle size by Zulfiqar et al. [13], this study adopted the 3:1 ratio of ammonia and ethanol in synthesizing the nanosilica particles. Nucleation and growth control are important conditions for the precipitation of monodisperse nanoparticles. In the case of a homogeneous solution, it is obtained by carefully determining the conditions that favour precipitation (concentration, additional rate of stabilizers and input chemicals, type, temperature and stoichimetry). Thus, the investigation of different concentration of sodium silicate solution: 0.5 N, 1 N, and 1.5 N. The synthesized nanosilica samples are presented in Figure 2.

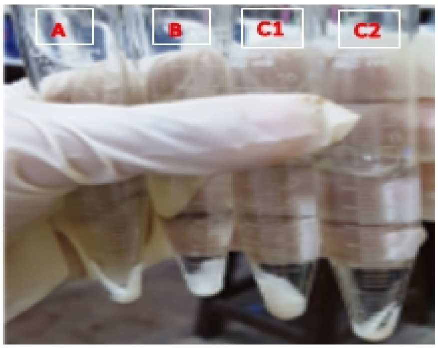

Figure 2. Precipitated Nanosilica for different Sodium Silicate Solution Concentration

Figure 2 shows different precipitated nanosilica samples: A, B, and C, from the different sodium silicate solution (SSS) concentrations 0.5N, 1N, and 1.5N respectively. The Sample A was prepared from SSS at 0.5 N, and the Sample B from SSS at 1 N; both were prepared with ultrasonic bath. The nanosilica from Sample C had two procedures: one with ultrasonic bath (with Procedure A, Sample C1) and other one without ultrasonic (with Procedure B, Sample C2). Table 3 shows the physical observations from the precipitated nanosilica particles.

Table 3. Samples of Silica Nanoparticles

Sample	**SSS**		**Distilled Water (ml)**	**Ammonia/ Ethanol Ratio**	**Observation**
	Conc. (N)	**Quantity (ml)**			
A	0.5	1	7	90/30	Few nanosilica particles were produced. They were dispersed and small.
B	1.0	2	7	90/30	More quantity of nanoparticles, apparently they were bigger than the former ones.
C	1.5	2	7	90/30	More quantity of silica nanoparticles. They were similar than the Sample B, but a little more agglomerated.

It was observed from SEM morphology in Figure 3, that the size of nanosilica in ammonia/ ethanol 3:1 ratio was large enough for application as water based mud system rheology enhancer additive. The literature shows that dilution of sodium silicate with water raised the volume of existing silicon-containing complexes that act as colloids. With the application of water, these neutral complexes were first broken down into sodium ions and complexes, and then condensed into larger colloids [14, 15]. The incorporation of ethanol and ammonia into dilute sodium silicate can lead to the formation of intermediate Si $(OH)_4$, followed by condensation in the form = Si-O-Si = [13]. This Si–O–Si was observed in the SEM analysis.

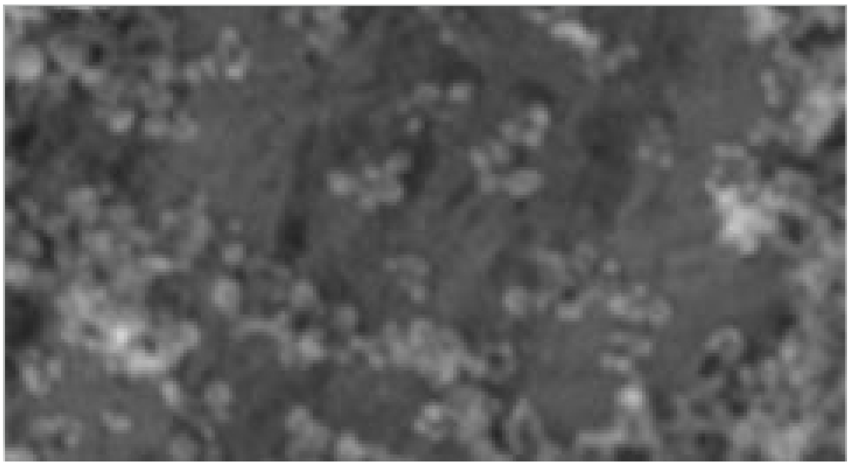

Figure 3. SEM image of nanosilica prepared in ammonia/ ethanol 3:1 ratio

3.2 Application of the Synthesized Nanosilica as Water Based Mud Additive

Three (3) water based mud system was formulated in this study, sample 1 is a standard water based mud system without nanosilica, sample 2 is standard water based mud system treated with 1 g of nanosilica, and sample 3 is standard water based mud system treated with 2 g of nanosilica. The flow properties which must remain stable when exposed to subsurface environment during drilling operation were examined. The Fann Viscometer was used to estimate the gel strength, plastic viscosity, apparent viscosity, and yield point of the formulated mud system samples. Knowing these parameters provides the information needed for daily monitoring of flow characteristics in a drilling system. Figure 4 shows that when 2 g of synthesized nanosilica was added to a standard water-based mud system, the mud system density increased. Certain changes in the composition affect the flow properties of the drilling fluid system. To prevent the possible occurrence of kick, the pressure of the mud column must be greater than the formation pressure, and in the empirical formula used in estimating the mud column; density is the only adjustable parameter. Therefore, it is useful to increase the density of the drilling fluid system if it is not in excess to cause formation fracture. The density of the drilling fluid also helps maintain the stability of the drilling site during drilling. The buoyant effect of drilling mud on drilling formation cuttings increases with increasing density, and this facilitates the transport of cuttings from the wellbore.

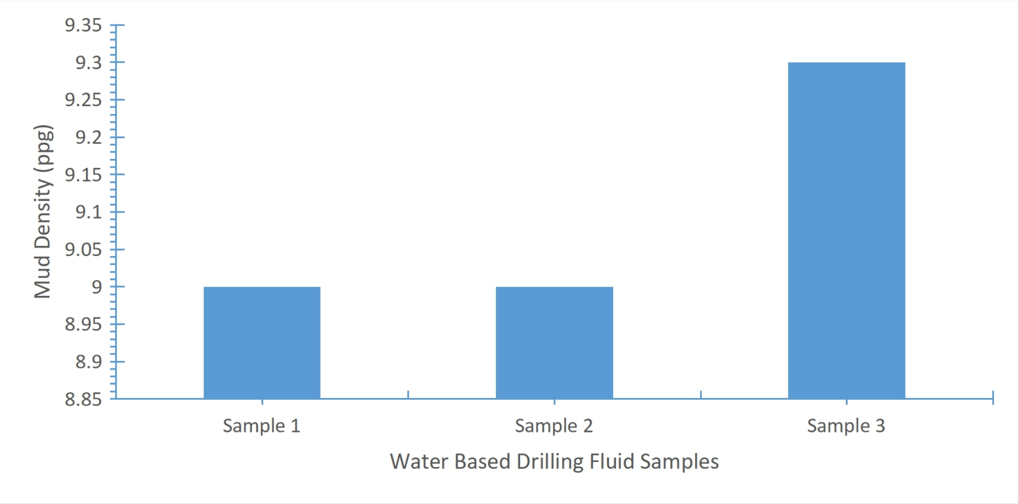

Figure 4. Formulated Mud System Samples Density

The flow characteristics of the drilling fluid play an important role in the success of the drilling process. These features are primarily responsible for the removal of cuttings, but also affect the drilling progress in many other ways. Figure 5 shows the flow characteristics investigated in this study. The values for plastic viscosity (PV), apparent viscosity (AP), and yield point (YP) are in line with the standards of the American Petroleum Institute (API). PV depends primarily on the amount of solids in the drilling fluid and the viscosity of the fluid. YP depends more on contamination with colloidal clay and inorganic salts, but in this study, the gel nature of nanosilica contributed to YP values.

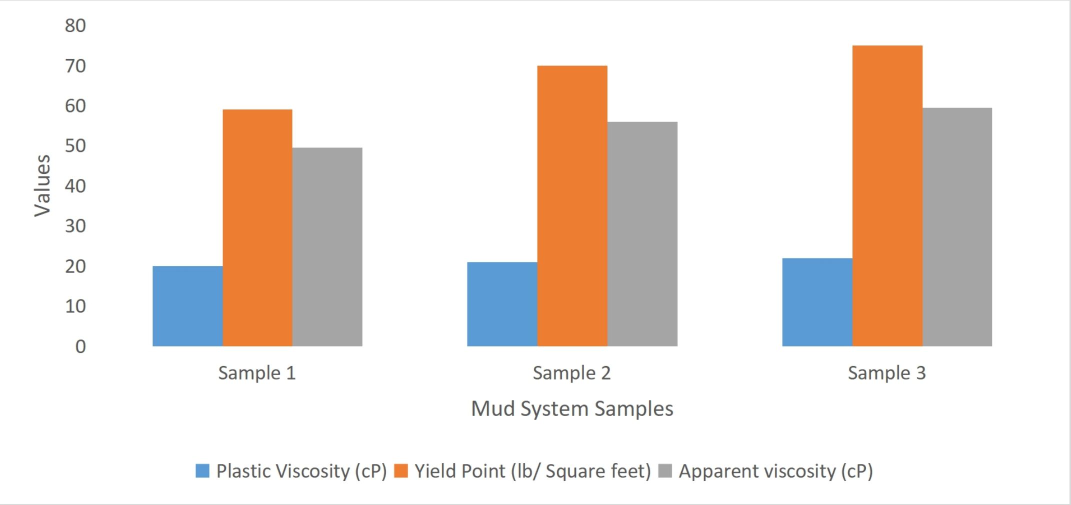

Figure 5. Estimated Flow Characteristics of the Water Based Mud Samples

The calculated Power Law flow behaviour index (n) using equation (1) shows that the values of “n” for the three mud system samples were less than one (Table 4). Thus, they are non-Newtonian fluids.

$$n = 3.32 log \frac{\theta_{600}}{\theta_{300}} \quad (1)$$

Table 4. Flow Behaviour Index, n for the formulated Mud System Samples

Mud System Samples	Flow Behaviour Index, n (Dimensionless)
1	0.3254
2	0.2994
3	0.2947

4. Conclusion

In this study, nanosilica was synthesized and the choice of this nanoparticle is related to its excellent chemical and physical properties which has a positive effect in the drilling fluids flow properties. There are different mechanisms for synthesizing nanosilica, but colloidal method was adopted to enable us have the nanosilica in a powder form for ease of application and storage. Sodium silicate solution (SSS), a cheap precursor was used to synthesize nanosilica. The Sodium Silicate Solution (SSS) was prepared at different concentrations: 0.5 N, 1 N, and 1.5 N, and the nanosilica synthesis carried out in the mixture of 3:1 ammonia/ ethanol ratio. Nanosilica synthesized with 1 N concentration of SSS gave the optimum size and quantity, and was used to formulate the examined high-performance water based mud system. The nanosilica had a positive effect for the flow properties of the drilling fluids samples.

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