# The Novel Approach for the Improvement of Fluid Loss Control in Water-based Mud using Nanosilica as Filler for Pectin

Ling Hua Sid

*Department of Petroleum Engineering,*
*Faculty of Chemical & Energy Engineering,*
*Universiti Teknologi Malaysia, 81310 Johor Bahru, Malaysia*

Issham Ismail

*Malaysia Petroleum Resources Corporation Institute for Oil and Gas,*
*Universiti Teknologi Malaysia, 81310 Johor Bahru, Malaysia*

Ariffin Samsuri

*Department of Petroleum Engineering,*
*Faculty of Chemical & Energy Engineering,*
*Universiti Teknologi Malaysia, 81310 Johor Bahru, Malaysia*

Zainal Zakaria

*Department of Petroleum Engineering,*
*Faculty of Chemical & Energy Engineering,*
*Universiti Teknologi Malaysia, 81310 Johor Bahru, Malaysia*

**Abstract - Fluid loss is the leakage of the liquid phase of drilling fluid, slurry or treatment fluid containing solid particles into the formation matrix. The resulting buildup of solid material or filter cake may be undesirable, as may the penetration of filtrate through the formation. Nanosilica and pectin are potential fluid loss additives used in water-based mud to reduce fluid loss and invasion of solid particles into the formation. The objective of this research work was to introduce nanosilica and pectin as fluid loss control agents in basic water-based mud at ambient condition and 250°F. The experimental results were then compared with commercial fluid loss control agent (i.e., HydroPac R) in basic water-based mud (WBM). The laboratory experiments were conducted using different concentrations ranging from 0.50 – 1.50 lb/bbl (wt. %) of nanosilica, pectin, and HydroPac R. The laboratory works were conducted as per the API Recommended Practice 13 B-1 (2009). The experimental results revealed that pectin gave a comparable fluid loss control performance as compared to HydroPac R (i.e., pectin was able to yield 7 cc of filtrate volume compared with HydroPac R which yield 6.6 cc of filtrate volume at the end of 30 minutes under low pressure of 100 psi and at ambient temperature) while nanosilica, an amorphous silica powder, produced the worst performance (i.e., 10 cc of filtrate volume at the end of 30 minutes). The experiment work was then repeated using the mixture of pectin and nanosilica as filler. The mixture yields a better reading than mud samples containing only pectin by reducing further fluid loss by 5%. The research work revealed that pectin with nanosilica as filler has the potential to be used as a fluid loss control agent in water-based mud as it produces a comparable performance with HydroPac R.**

KEYWORDS: FLUID LOSS, FLUID LOSS CONTROL AGENT, NANOSILICA, PECTIN, WATER-BASED MUD

## I. INTRODUCTION

Fluid loss may occur during drilling, cementing and completion job. It refers to the liquid phase of drilling mud that enters the formation when hydrostatic pressure in the hole is higher than formation pressure. At the same time, the solid particles in the drilling mud may invade the porous formation. Fluid loss may cause many drilling problems. Additives are added into the drilling mud to combat fluid loss into the formation. In this research, nanosilica and pectin are used as the additives to improve fluid loss.

Nanosilica are commonly defined as objects with a diameter in range of 1 to 100 nanometer (nm). To be exact, the size of nano-sized silica is 30 nm (Y. H. Lai, 2007). Nanosilica exhibits properties such as being lightweight, ultrahigh strength, high electrical and heat conductivity and an increased surface area. Nature of nanomaterial very fine having high specific surface area with large area interactions only require very low concentration to enhance the properties of WBM (A. A. Ismail, 2016). Nanosilica is not new, but its application in the oil and gas industry certainly in its infancy, including drilling applications (N. Nabhani, 2012). Nanosilica is good in preventing shale swelling (H. Pham, 2014) because the pore throat size of shale ranges from 0.1 to 0.005 µm (P. H. Nelson, 2009). The nanosilica are small enough to penetrate and seal the pore throats in shale (C. Ma, 2013), and built internal mud cake, resulting in the reduction of fluid penetration into the shale. Because of high costs and greater risk of adapting new technologies, the application in oil and gas industry has not been fully discovered. However, the quest for efficiency in the current economic situation has been the drilling force behind development of new technologies.

Pectin is a soluble gelatinous polysaccharide presents in primary cell wall and middle lamella of terrestrial plants and fruit. Pectin can be extracted from fruits such as grapes, apples and citrus fruits. Pectin has the ability to form gels. The important keys in the aggregation of pectin molecules are hydrogen bonding and hydrophobic interaction. Hydrogen bonds with free hydroxyl groups on the molecules cause the water trapped into its three dimensional pectin network.

## II.    METHODOLOGY

The method to prepare pectin can be sourced from the technical paper entitled "Extraction and Characterization of Pectin from Passion Fruit Peels" (S. Q. Liew, 2014) and the method to prepare nanosilica can be sourced from the technical paper entitled "Extraction and Characterization of Nanosilica from rice husk" (V. B. Carmona, 2013).



Figure 1 Nanosilica



Figure 2 Pectin



Figure 3 HydroPac R

In this research work, the performances of nanosilica (Figure 1) and pectin (Figure 2) in water-based mud (WBM) were compared with the commercial fluid loss control agent (i.e., HydroPac R in Figure 3). The basic WBM formulation was obtained from Scomi Energy, as shown in Table 1, while the rheological and filtration test on the mud samples before and after aging were conducted as per the API RP 13B-1(2009). The rheological properties measured were mud density, plastic viscosity, apparent viscosity, yield point, gel strength at 10 second and 10 minutes using a rheometer. The research was finalized by mud filtration for 30 minutes at low pressure of 100 psi and ambient temperature.

Table 1 Basic water-based mud formulation

| Additives | Quantity (lb/bbl) |
|---|---|
| Bentonite | 15.0 |
| Soda ash | 0.25 |
| Caustic soda | 0.25 |
| Starch | 1.00 |

Density of the mud samples were kept constant at 10 ppg. To obtain the constant mud density, barite was added to drilling mud based on the desired amount resulted from calculations. The nanosilica first need to be dispersed for 1-2 hours before mixed with WBM. It was prepared using the concentrations by weight of 0.50 lb/bbl, 0.75 lb/bbl, 1.00 lb/bbl, 1.25 lb/bbl, 1.50 lb/bbl and those mud samples were tested for their rheological properties and filtration properties. The aging test was conducted at 250°F using only the optimum concentration of nanosilica at ambient

temperature. The same experimental procedures were repeated for pectin and HydroPac R. Noted that the aging cell was required to be taken out after 16 hours from the roller oven, cooled, and the degraded samples were tested for its rheological properties and filtration properties.

*Dispersion of Nanosilica*
Nanosilica must be dispersed first before adding into WBM. The desired concentration of nanosilica was dispersed in a 100 ml reagent bottle with distilled water of 50 ml (Figure 4). The reagent bottle was then ultra-sonicated for 1-2 hours depending on the amount of nanosilica added. It has to be dispersed until homogenous dispersion of nanosilica can be seen.



Figure 4 Dispersion using a reagent bottle

*Preparation of Composite*
The composite mixture of pectin and nanosilica was prepared with the proportion as per Table 2.

Table 2 Proportions of composite mixture

| Composite | |
|---|---|
| Pectin (lb/bbl) | Nanosilica (lb/bbl) |
| 0.48 | 0.02 |
| 0.71 | 0.04 |
| 0.94 | 0.06 |
| 1.17 | 0.08 |
| 1.40 | 0.10 |

The composite mixture samples were then tested for their rheological and filtration properties like the usual normal procedure previously. Figure 5 shows the flowchart for the accomplished laboratory works.
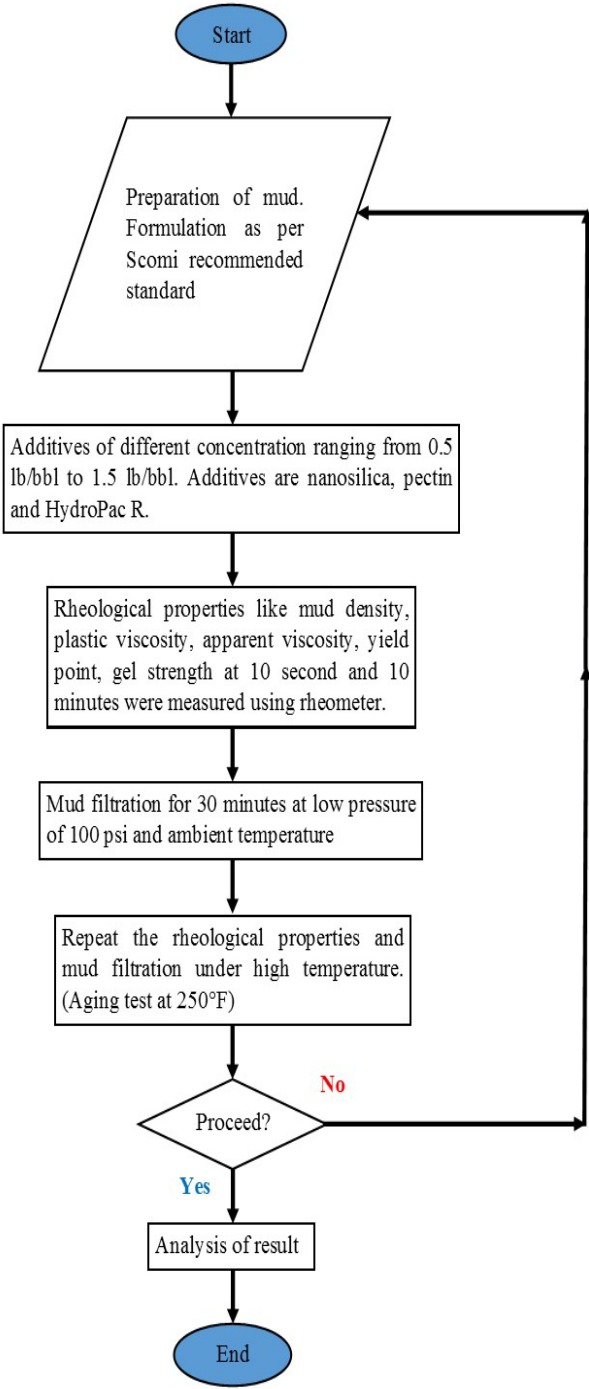
Figure 5 Flowchart for the laboratory works
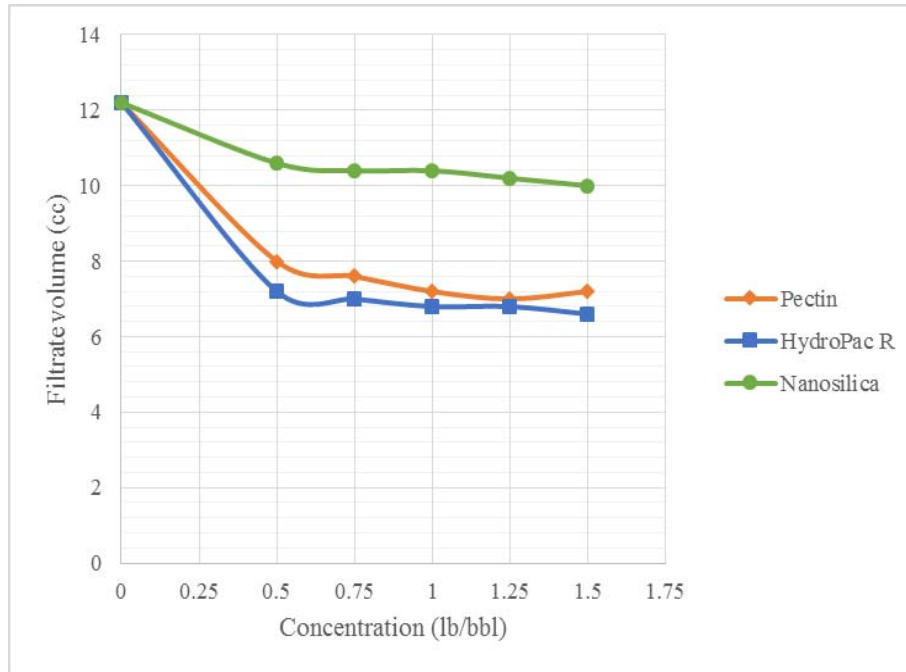
III.        EXPERIMENT AND RESULT

Figure 6 Fluid loss for nanosilica, HydroPac R, and pectin with concentrations
ranging from 0-1.5 lb/bbl at 30th minute

The filtration test experiment was carried out for 30 minutes. In Figure 6, the curves showed that the filtrate volume was influenced by the concentration of each of the fluid loss control agents. Increase in concentration of fluid loss control agent would reduce the volume of filtration. The lowest filtrate volume obtainable for nanosilica, pectin and HydroPac R are 10 cc, 7cc and 6.6 cc respectively. The optimum concentration of fluid loss control agent should be determined in order to study the optimum performances. It can clearly be seen here that the optimum concentration is 0.50 lb/bbl. Further increase in concentrations doesn't economically serve its objective because the cost surpasses the benefits. Therefore, this concentration of fluid loss additives (0.50 lb/bbl) was used for the following mud testing. At this optimum concentration, the filtrate volume of sample containing pectin and HydroPac R were 8 cc and 7.2 cc respectively while nanosilica was 10.6 cc. At this temperature, nanosilica reduced fluid loss by 13.11%, HydroPac R reduced fluid loss by 40.98%, pectin reduced fluid loss by 34.43%. The filtrate volume of nanosilica, HydroPac R and pectin were lower than the filtrate volume of WBM. In comparison among fluid loss control agents, the results revealed that nanosilica performed the worst compared to pectin and HydroPac R. Meanhile, HydroPac R performed slightly better than pectin. From here, we can see that pectin rival with the performances of the commercial HydroPac R.

The results can be explained in the following manner. Pectin is a cellulose material that can trap water particle into its three dimensional network and HydroPac R is a polyanionic cellulose which acts as a thickener in low-density brines (Scomi, 2013). HydroPac R is a cellulose derivative similar in structure, properties and usage in drilling fluids to carboxymethyl cellulose which contains high OH groups of negative charge. This negative charge tends to form hydrogen and hydroxyl bonding. The main reason nanosilica does not perform as much as pectin and HydroPac R probably because the size too small that almost all the nanosilica flowed out together with the filtrate causing almost no effect to the fluid loss control. The filter paper pore size is 60 µm.
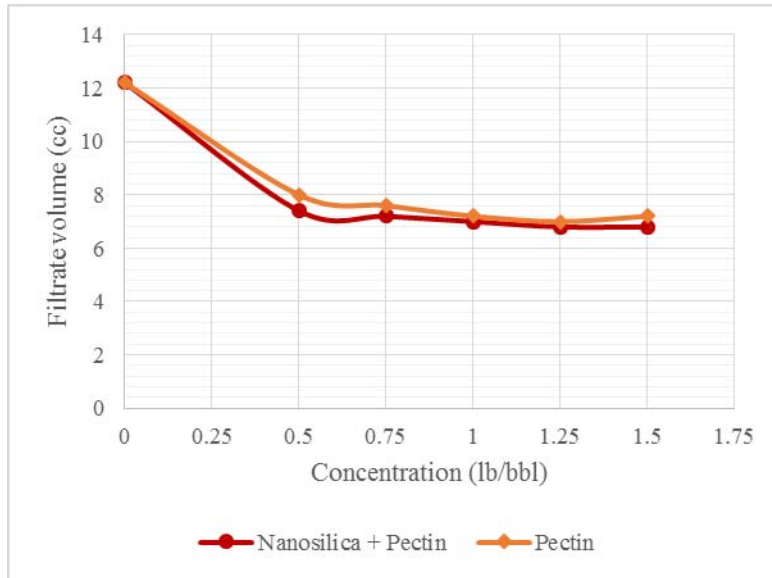
Figure 7 Fluid loss comparison for pectin and mixture of
pectin and nanosilica

The experiment was then repeated again using composite mixture of pectin and nanosilica. It is believed that nanosilica can be the filler in between the larger pectin molecules. Based on Figure 7, we can clearly see that at the optimum concentration, the filtrate loss in composite mixture sample perform better than the mud sample containing only pectin. To be exact, the composite mixture sample reduced extra of 5% fluid compared to the individual performance. From here, we can infer that nanosilica has the potential to be filler in between the pectin molecule to form a better composite fluid loss control additive. Since both exhibit the properties of hydrophilic, they are most likely to form mixture together without any agglomerations.



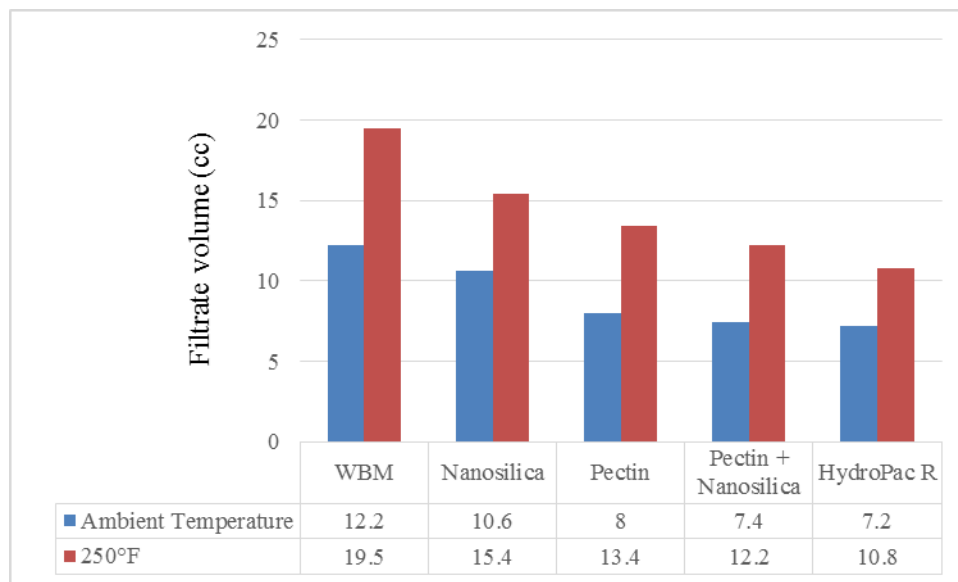| | WBM | Nanosilica | Pectin | Pectin + Nanosilica | HydroPac R |
|---|---|---|---|---|---|
| ■ Ambient Temperature | 12.2 | 10.6 | 8 | 7.4 | 7.2 |
| ■ 250°F | 19.5 | 15.4 | 13.4 | 12.2 | 10.8 |

Figure 8 Fluid loss at 30th minutes before and after aging (0.5 lb/bbl weight %)

Figure 8 shows the same trend that the nanosilica, pectin, composite mixture and HydroPac R performed better in ambient temperature than in high temperature (250°F). Nanosilica, pectin, composite mixture and HydroPac R loss extra 45.28%, 67.5%, 64.86% and 50% fluid respectively compared to their initial performances at ambient temperature. At high temperature, the mud samples degraded and caused the fluid loss became more than the previous one. From the data given above, we can inferred that the composite mixture actually helps better in

reducing any further fluid loss in both ambient and high temperature condition since it has a low filtrate volume which is comparable to HydroPac R. HydroPac R was taken as a benchmark to compare the other result. Somehow, overall they showed a decrease trend in performances when exposed to high temperature.



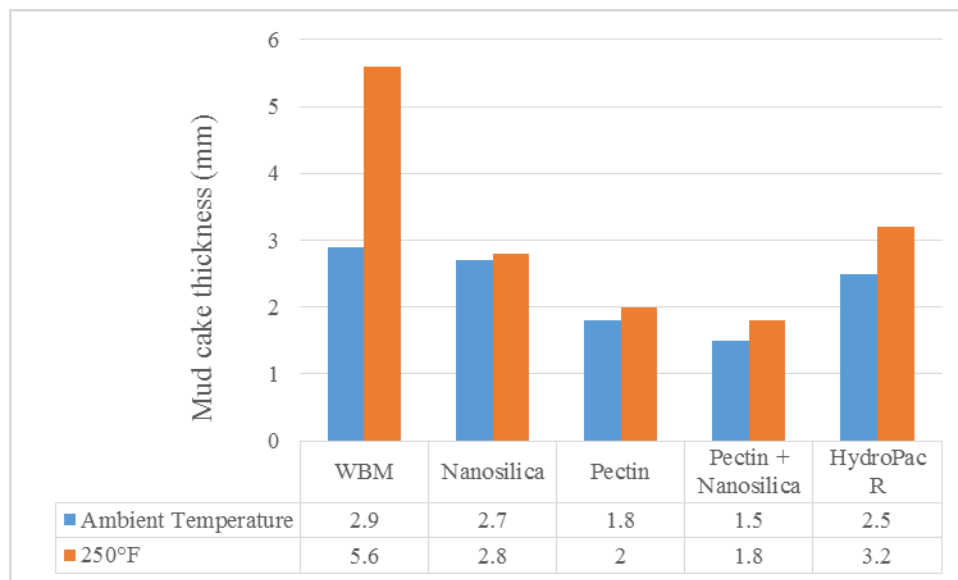| | WBM | Nanosilica | Pectin | Pectin + Nanosilica | HydroPac R |
|---|---|---|---|---|---|
| ■ Ambient Temperature | 2.9 | 2.7 | 1.8 | 1.5 | 2.5 |
| ■ 250°F | 5.6 | 2.8 | 2 | 1.8 | 3.2 |

Figure 9 Mud cake thickness of all samples before and after aging (0.5 lb/bbl weight %)

Figure 9 shows the mud cake thickness for each sample containing different fluid loss control agent and WBM at optimum concentration. At ambient temperature, the mud cake formed by sample containing WBM was the thickest indicated most permeable among all the mud samples. The mud cake formed by sample containing composite mixture was the thinnest and the least permeable compared to other mud samples. Fluid loss control agent must be able to control fluid loss and the formation of mud cake effectively. The filtrate volume should be low, the mud cake thickness and its permeability must be low (Ryen, 2011). Meanwhile at temperature of 250°F, the thickest mud cake was WBM and the thinnest mud cake was also the sample containing composite mixture. Thickest mud cake indicated the highest permeability and the thinnest mud cake indicated the lowest permeability. The mud cake formed with the low permeability prevents fluid loss and invasion of solid particles into the formation by closing the pore spaces on the surface of formation. From here we could inferred that mud cake formed by sample containing composite mixture before aging and after aging also maintained as the thinnest mud cake among all the samples. In terms of individual performances, pectin perform the best compared to the other fluid loss control agent but somehow, with the addition of nanosilica forming composite showed an even better result compare to mud sample of pectin alone. Pectin might be a good fluid loss control agent because it form thin mud cake which is desirable in the industry. The nanosilica does not increase much of its mud cake thickness compared to its previous one at ambient temperature. Nanosilica does not perform well alone but with the help of pectin, it can form a better composite which is better than pectin alone.
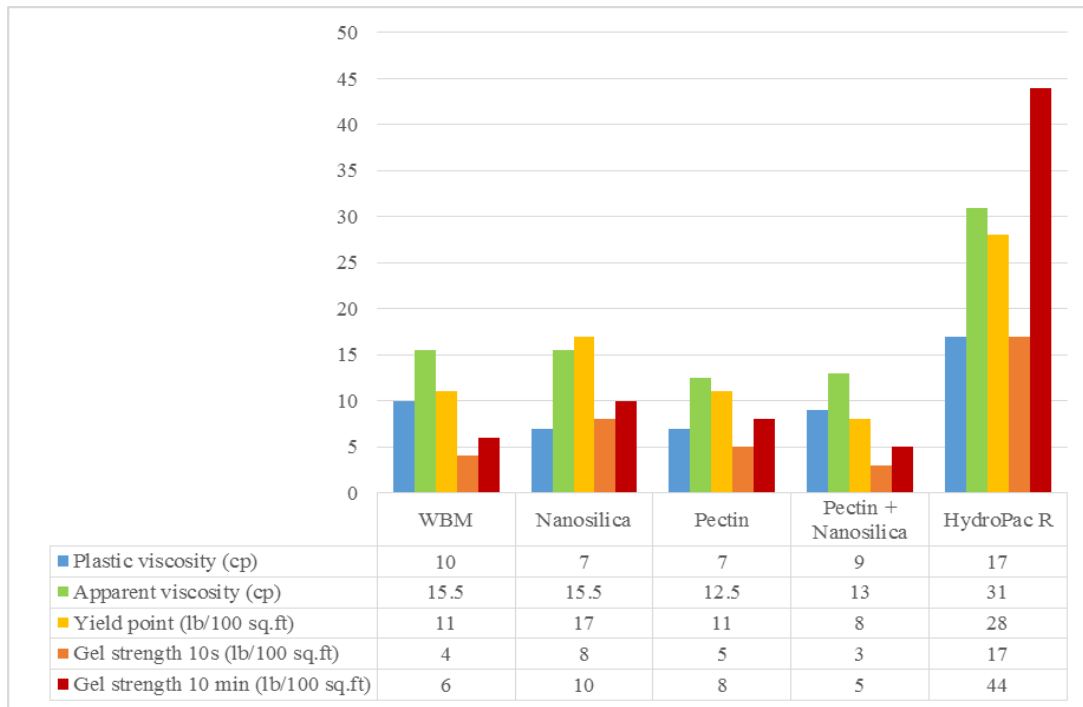
| | WBM | Nanosilica | Pectin | Pectin + Nanosilica | HydroPac R |
|---|---|---|---|---|---|
| ■ Plastic viscosity (cp) | 10 | 7 | 7 | 9 | 17 |
| ■ Apparent viscosity (cp) | 15.5 | 15.5 | 12.5 | 13 | 31 |
| ■ Yield point (lb/100 sq.ft) | 11 | 17 | 11 | 8 | 28 |
| ■ Gel strength 10s (lb/100 sq.ft) | 4 | 8 | 5 | 3 | 17 |
| ■ Gel strength 10 min (lb/100 sq.ft) | 6 | 10 | 8 | 5 | 44 |

Figure 10 Rheological properties of all the mud samples (0.5 lb/bbl weight%) before aging



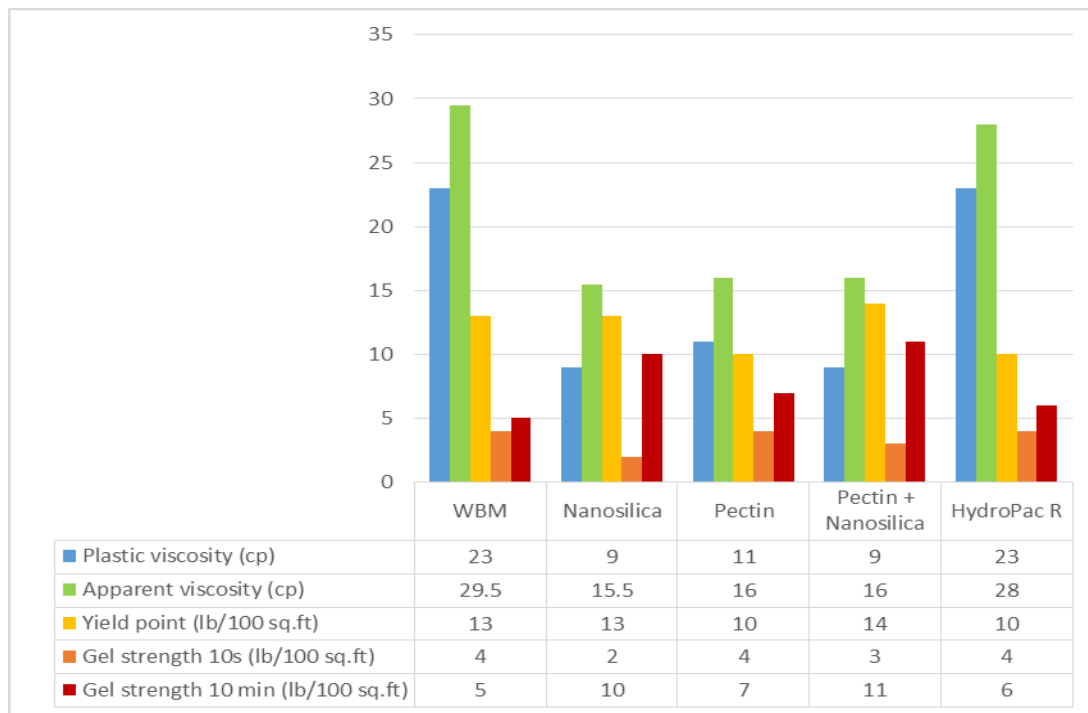| | WBM | Nanosilica | Pectin | Pectin + Nanosilica | HydroPac R |
|---|---|---|---|---|---|
| ■ Plastic viscosity (cp) | 23 | 9 | 11 | 9 | 23 |
| ■ Apparent viscosity (cp) | 29.5 | 15.5 | 16 | 16 | 28 |
| ■ Yield point (lb/100 sq.ft) | 13 | 13 | 10 | 14 | 10 |
| ■ Gel strength 10s (lb/100 sq.ft) | 4 | 2 | 4 | 3 | 4 |
| ■ Gel strength 10 min (lb/100 sq.ft) | 5 | 10 | 7 | 11 | 6 |

Figure 11 Rheological properties of all the mud samples (0.5 lb/bbl weight%) after aging

Viscosity is the measurements related to the flow of the liquids. As the fluid loss control agent was added into WBM, the rheological properties underwent changes. In Figure 10, the viscosity of HydroPac R was the highest followed by WBM, composite mixture, nanosilica and pectin with the same magnitude. In general, the rheological properties of the mud changed as the temperature increased. Makinde (2010) stated that the viscosity and yield point decreased steadily with increased in temperature. In Figure 11, the viscosity of WBM and HydroPac R were the

highest followed by pectin, nanosilica and composite mixture with the same magnitude. Based on Scomi (2013), recommended viscosity of WBM should be less than 25 cp.

Yield point is a measurement of ability of the mud to lift cuttings out of annulus of borehole and said to be as attractive forces among colloidal particles in drilling mud. In Figure 10, the yield point of HydroPac R was the highest followed by nanosilica, pectin and WBM with the same magnitude, and the composite mixture was the least yield point. The yield point of composite mixture was the highest followed by WBM and nanosilica with the same magnitude, pectin and HydroPac R have the least yield point. Based on Scomi (2013), recommended value for yield point of WBM is more than 25 lb/100 sq.ft. High yield point mud carry cuttings better than a low yield point mud of similar density. Yield point of all the fluid loss control agents decreased at high temperature because degradation of solid particles and the expansion of the molecular distance lower the resistance of the fluid to flow (Amani, 2012).

Gel strength is defined as a measure of ability of colloidal dispersion to develop and retain a gel form, which based on its resistance to shear. The gel strength determines the ability of the mud to hold solids in suspension. Gel strength of 10 second in Figure 10, the highest was HydroPac R followed by nanosilica, pectin, WBM and composite mixture. In Figure 11, the highest yield point were WBM, pectin, HydroPac R with the same magnitude followed by composite mixture and nanosilica. Gel strength of 10 minutes in Figure 11, the highest was HydroPac R followed by nanosilica, pectin, WBM and composite mixture. In Figure 11, the highest was composite mixture followed by nanosilica, pectin, HydroPac R and WBM has the least gel strength. Thermal degradation of all the mud samples after aging (250°F) caused its gel strength to reduce. If the mud has a high gel strength, it needs a high pump pressure in order to break the circulation after the mud has been in static condition for a long time. Recommended gel strength by Scomi (2013) is 7-9 lb/100 sq ft.

## IV. CONCLUSION

Nanosilica performed the worst compared to pectin and HydroPac R. Pectin can be used as an alternative to replace HydroPac R as fluid loss control agent in the industry. Pectin together with the WBM formulation gave a positive results like HydroPac R because it produces desirable low filtrate volume and thin mud cake. Since nanosilica does not perform as expected individually, it can be added into basic water-based mud with pectin mixture as filler between the bigger pectin molecules to control better the fluid loss. Nanosilica only needs a small concentration to enhance the properties.

## V. RECOMMENDATION

Further research works are required to improve nanosilica as fluid loss control agent since it has a lot of advantages. Studies on performance of pectin and nanosilica under high pressure and high temperature (i.e., 500 psi and 250°F) are required. To achieve better performance of rheological properties, researchers may consider using other mud formulations that include xanthan gum and KCL.

REFERENCES

[1]   A. Ismail, T. C. Seong, N. A. Buang, W. R. Wan Sulaiman (2014). "Improve performance of water-based drilling fluids using nanoparticles". Petroleum Engineering Department, Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia.
[2]   Aftab, A. R. Ismail, Z. H. Ibupoto (2016). "Enhancing the rheological properties and shale inhibition behavior of water-based mud using nanosilica, multi-walled carbon nanotube, and graphene nanopatelet". Petroleum and Natural Gas Engineering Department, Mehran UETSZAB, Sindh, Pakistan.
[3]   Sameni, P. Pourafshary, M. Ghanbarzadeh, S. Ayatollahi (2015). "Effect of nanoparticles on clay swelling and migration". Institute Petroleum of Engineering, University of Tehran, Iran.
[4]   M. Salem Ragab, A. Noah (2014). "Reduction of formation damage and fluid loss using nano-sized silica drilling fluids". PhD Thesis. American University in Cairo and Suez University, Egypt.
[5]   R. Ismail, A. Aftab, Z. H. Ibupoto, N. Zolkifile (2016). "The novel approach for the enhancement of rheological properties of water-based drilling fluids by using multi-walled carbon nanotube, nanosilica and glass beads". Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia.
[6]   R. Ismail, W. R. Wan Sulaiman, M. Z. Jaafar, I. Ismail, E. S. Hera (2016). "Nanoparticles performance as Fluid Loss Additives in Water Based Drilling Fluids", Materials Science Forum, Vol. 864, pp 189-193. Petroleum Engineering Department, Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia.
[7]   American Petroleum Institute (2009). *Recommended Practice 13B: Recommended Practice for Field Testing Water-Based Drilling Fluids*. Dallas: American Petroleum Institute.

[8]  Fazelabdolabadi, A. A. Khodadadi (2015). "Thermal and rheological properties improvement of drilling fluids using functionalized carbon nanotubes". Petroleum Engineering Research Division, Research Institude of Petroleum Industry (RIPI), West Blvd. Azadi Sports Complex, P.O. Box 14665-1998, Tehran, Iran.

[9]  Ma, L. LI, S. LI, X. Han (2013). "Application of nanomaterials in the fields of drilling fluids and completion fluids", Key Engineering Materials, Vols. 562-565, pp 576-580. Jiangxi Science and Technology Normal University, Nanchang 330013, P. R. China.

[10] Kasiralvalad (2014). "The great potential of nanomaterials in drilling & drilling fluid applications". Science and Research Branch, Islamic Azad University, Faculty of Petroleum Engineering, Daneshgah Blvd, Simon Bulivar Blvd, Tehran, Iran.

[11] H. Pham, Q. P. Nguyen (2014). "Effect of silica nanoparticles on clay swelling and aqueous stability of nanoparticle dispersions". Petroleum and Geosystems Engineering Department, The University of Texas at Austin, 200 E. Dean Keeton, Austin, TX 78712-1061 USA.

[12] Ismail, A. B. Mohd. Nor, M. F. Hamid, A. R. Ismail (2015). "The impact of durian rind in water-based mud in combating lost circulation". Malaysia Petroleum Resources Corporation Institute for Oil and Gas, Universiti Teknologi Malaysia, 81310 Johor Bahru, Malaysia

[13] J. Nasser, A. Jesil, T. Mohiuddin, M. Al Ruqesh, G. Devi, S. Mohataram (2013). "Experimental Investigation of Drilling Fluid Performance as Nanoparticle". Mechanical & Industrial Engineering Department, Caledonian College of Engineering, Muscat, Oman.

[14] J. P. Deville, B. Fritz, M. Jarret (2011). "Development of water-based drilling fluids customized for shale reservoirs." SPE Drilling and Completions.

[15] K. Song, Q. Wu, M. Li, S. Ren, L. Dong, X. Zhang, T. Lei, Y. Kojima (2016). "Water-based bentonite drilling fluids modified by novel biopolymer for minimizing fluid loss and formation damage". School of Renewable Natural Resources, Louisiana State University, Baton Rouge, LA 70803 USA.

[16] M. S. Al-Yasiri, W. T. Al-Sallami (2015). "How the drilling ffluids can be made more efficient by using nanomaterials". Chemical Engineering Deparment, College of Engineering, University of Baghdad, Iraq.

[17] M. D. Adejumo, C. T, Ako, V. E. Efeovbokhan (2010). "Modelling the temperature and aging time on the rheological properties of drilling effect of temperature fluids". Department of Petroleum Engineering, Covenant University, Nigeria.

[18] N. Alias, N. F. M. Tahir, T. A. T Mohd, N. A. Ghazali, E. Yahya, M. Z. Shahruddin, N. A. Ramlee, A. Azizi (2014). "Rheological study of nanosilica based drilling fluid", Applied Mechanics and Materials, Vol. 575, pp 128-133. Faculty of Chemical Engineering, Universiti Teknologi Mara, 40500 Shah Alam, Selangor, Malaysia.

[19] N. Nabhani, M. Emami (2012). "The potenial impact of nanomaterials in oil drilling industry". Petroleum University of Technology, nabhani@put.ac.ir.

[20] N. S. Al-Zubaidi, A. A. Alwasiti, D. Mahmood (2016). "A comparison of nano entonite and some nano chemical additives to improve drilling fluid using local clay and commercial bentonites". Department of Petroleum Engineering, College of Engineering, University of Technology, Iraq.

[21] P. H. Nelson (2009). "Pore throat sizes in sandstones, tight sandstones, and shales." U.S. Geological Survey.

[22] R. Caenn, H. C. H, Darley, G. R. Cray (2011). *Composition and properties of drilling and completion fluids*. 6th ed. Houston: Gulf Professional Publishing.

[23] R. F. Sigal (2015). "Pore-size disributions for organic-shale-reservoir rocks from nuclear-magnetic-resonance spectra combined with adsorption measurements". Society of Petroleum Engineers.

[24] S. Agarwal, P. Tran, Y. Soong, D. Martello, R. K. Gupt (2015). "Research shows benefits of adding nanoclay, nanosilica to oil-based hp/ht drilling fluids". *The American Oil & Gas Reporter*.

[25] S. S. Hassani, A. Amrollahi, A. Rashidi, M. Soleymani, S. Rayatdoost (2016). "The Effect of nanoparticle on the heat transfer properties of drilling fluids". Nanotechnology Research Center, Research Institude of Petroleum Industry (RIPI), P.O. Box 14665-137, Tehran, Iran.

[26] S. Q. Liew, N. L. Chin, Y. A. Yusof (2014). "extraction and characterization of pectin from passion fruit peels". Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia.

[27] B. Carmona, R. M. Oliveira, W. T. L. Silva, L. H. C. Mattoso, J. M. Marconcini (2013). "Nanosilica from rice husk: Extraction and characterization". Departamento de Engenharia dos Materiais, Universidade Federal de Sao Carlos, SP, Brazil.

[28] Y. H. Lai, M. C. Kuo, J. C. Huang, M. Chen (2007). "thermomechanical properties of nanosilica reinforced PEEK composites", Key Engineering Materials Vol. 351 (2007) pp. 15-20. Institute of Materials Science and Engineering; Center for Nanoscience and Nanotechnology, National Sun Yat-Sen University, Kaohsiung, Taiwan 804, R. O. China.