Effect of temperature and concentration of industrial waste graphene on rheological properties of water based mud

Putri Humairah Monashofian Putra, Badrul Mohamed Jan, Mohamad Fazly Abdul Patah and Mohd Usman Mohd Junaidi
Department of Chemical Engineering, Faculty of Engineering, University of Malaya, Kuala Lumpur, 50603, Malaysia

Abstract. It is no secret that nano technology has been widely applied in various industries. In the upstream oil and gas industry, nanoparticle technology has received widespread attention due to its ability to enhance drilling fluid properties; thus improves drilling efficiency and reduces the overall operation cost. The objective of the study is to investigate the effects of temperature and concentration of graphene retrieved from unwanted industrial waste on rheological and filtration properties of the water based drilling mud (WBM). It is known that graphene is very expensive. At the same time, the availability and abundance of industrial carbon based waste presents an opportunity to extract graphene from unwanted, inexpensive waste and utilise it to improve the drilling fluid properties. Three types of drilling fluids are formulated (basic WBM, WBM plus commercial graphene and WBM plus waste graphene) and tested based on the recommended API 13B-1 practice. Both commercial and waste graphene concentration was varied at 0.05, 0.1 and 0.2 ppb. For each concentration, the mud rheological and filtration properties were measured before and after aging for 16 hours at different temperature of 200°F, 225°F and 250°F while the pressure was fixed at 500 psi. Results showed that the addition of small concentration of graphene nanoparticles increases the plastic viscosity (PV), yield point (YP), and improved the API and HPHT filtrate loss volume. However further increment in concentration caused the filtration property to deteriorate hence 0.05 ppb was taken as the optimum amount. It reduced the filtration loss by 8.43% at API condition and 18.57%, 13.82% and 14.06% at 200°F, 225°F and 250°F respectively.

Keywords: water based drilling fluid; industrial waste; graphene; rheological properties; filtration control

1 Introduction
1.1 Background and rationale of study
The oil and gas industry is a multi-billion dollar market that is not likely to go away any time soon. Despite the renewable energy alternatives, they still face technological inertia and will take years to revolutionise (White, 2008). Through 2040, half of the global energy demand is expected to be satisfied mainly by fossil fuels. However the aging of shallow boreholes and depletion of usual oil and gas resources is triggering the fossil fuel supply shortage. The most plausible solution to overcome this energy plight is to discover and bore more petroleum wells and harsh and deep reserves by 2020 (Aftab, Ismail, Ibupoto, Akeiber, & Malghani, 2017). Unfortunately this also means that drilling of reservoirs is going to be more intricate, challenging and costly as most of unconventional oil bores and geo-thermal wells happened at high pressure high temperature (HPHT) conditions.

The drilling operation is a critical stage in any well construction as it is virtually the most expensive phase in the oil and gas production. Drilling mud plays a crucial part in the drilling process as its performances influence the drilling efficiency, hence the overall cost of the operation. The base fluid
can either be water or oil, and is used to classify the drilling fluids, thus giving the name of water based mud (WBM) or oil based mud (OBM).

The primary functions of drilling fluids are to lift and carry the drilled solid pieces to the earth surface, clean and lubricate the wellbore, lower the drill bit temperature, maintain wellbore stability by governing the formation pressure, impede reservoir fluids influx from penetrated rocks and drilling fluid outflow to the reservoir by forming thin and low permeability filter cake (Caenn, Darley, & Gray, 2017). A good drilling fluid should be able to tolerate the downhole extreme HPHT condition. Improper selection and inappropriate formulation of the drilling mud may result in many drilling setbacks such as loss of drilling mud, blocked pipe and blowout which would interrupt and delay the drilling process and incur additional expenses.

![Figure 1. Schematic representation of drilling process (Zisis, Vryzas., & Vassilios, C. Kelessidi., 2017).](image)

Different type of drilling mud is used for different reservoirs as its selection is subject to geological condition, historical field data and ease of disposal after no longer in use. OBM exhibits better rheological performance and shale inhibition, but its usage is strictly restricted in certain areas because it causes high toxicity level, poor emulsion stability at HPHT and affects well logging operation. WBM is regarded as the cheapest and ecologically friendly mud; and can provide a safer option to bore water susceptible shale at lower disposal price besides showing better shale inhibition. As the world is becoming more environmentally conscious, it has become more critical than ever for the oil and gas industry to minimize the environmental footprints associated with oil recovery processes.

WBM consists of approximately 80% water (continuous phase) and 20% drilling additives and is employed to drill nearly 80% of all the world’s wells. The stabilizers and inhibitors contained in a conventional WBM are macro size and cannot seal the shale nanopores. Unfortunately due to its high water content, this normally leads to high filtrate loss, clay swelling and shale instability. At present, the drilling sector experiences technical drawback to design drilling muds with improved rheological properties at HTHP conditions. Nanoparticle provides a good remedy to plug the nano pore size of the shale. Hence the application of graphene nano materials from industrial waste is proposed to enhance the thermal and physical properties of a water- based mud (WBM) and overcome the shortcomings. Emerging nanotechnology involving graphene and graphene derivatives such as graphene oxide have promised good prospects in the petroleum upstream trade in the drilling, hydraulic fracturing, shale inhibition, rheology modification, and wellbore consolidation areas.

1.2 Problem statement
Despite the importance of graphene in various applications not limited to drilling but also energy storage and conversion, catalysis, and electronic fields, it is alas very expensive. Currently a 50 g of graphene is sold at around RM1,000. At the same time, the availability and abundance of industrial carbon based
waste triggers the initiatives to convert the waste into wealth. This presents an opportunity to extract graphene from unwanted inexpensive waste and utilise it to improve the drilling fluid properties. The purpose of the study is to investigate the effects of commercial graphene and graphene from industrial waste on rheological properties of the water based drilling mud (WBM).

1.3 Significance of study
Synthesising graphene from unwanted waste is the unique feature of this study as we add values to the waste and promote sustainability and green environment. A successful experimental study of this research will contribute to the enhancement of the drilling fluid behaviours, which will in turn lead to the proper handling of a well control system. This will ease the management of a drilling operation besides ultimately increasing the profitability of the oil and gas industry.

2 Critical literature review
2.1 Challenges of drilling industry
With the decline of conventional oil and gas reserves, the industrial players resort to explore deep, unconventional wells which are 4200 m below sea level. This adds to the complexity of the drilling operation as more than 50% of the present earth’s resource are at severe settings with temperature and pressure exceeding 200°C and 1600 bar respectively. When exposed to HTHP applications, the drilling fluids faced several main limitations including lesser stability, increase in fluid density and this every so often leads to earlier equipment wear and tear besides higher well treatment costs. Moreover, shale swelling caused by the interaction of clay minerals with water is a concern because it results in wellbore instability, caving and reduced mud efficiency to suspend the drilled cuttings.

Presently the greatest hindrance encountered by the oil and gas corporation is to design enhanced drilling fluids with improved rheological, filtration and well strengthening characteristics which can cope with the elevated bottom hole temperature and pressure over 350°F and 10,000 psi (Aftab, Ismail, & Ibupoto, 2017). With the growing demand for liquid fuel consumption and the diminishing amount of readily attainable oil in the forthcoming years, employing novel practices to the industry will be vital to meet the future global energy requirement. Nano-graphene in WBM is shown to have the potential to tackle such concerns in the industry.

2.2 An overview of graphene nanotechnology
Over the last decades nanotechnology has found its way in various fields including oil and gas industry. The functions of nanoparticles such as SiO2 and TiO2 as additives in drilling fluids formulation have been reported in numerous studies recent years (Parizad, Shahbazi, & Ayatizadeh Tanha, 2018; Rafati, Smith, Sharifi Haddad, Novara, & Hamidi, 2018). However, graphene only enjoys significant attention within the last 10 years or so after the pioneers, Prof. Andre K. Geim and Prof. Konstantin Novoselov won a Nobel Prize for successfully isolating graphene from graphite in 2007.

Graphene is described as a wonder material of the 21st-century that has opened up many possibilities for creating the world of tomorrow as it started to revolutionise every sector from health, energy to environment (Gorey, 2017). It is the thinnest compound known to man consisting of one single layer sp2 hybridized carbon atoms arrayed in a hexagonal honeycomb pattern. Its’ astounding properties such as high surface area (2620 m²/g), good chemical stability, high electrical conductivity and mechanical strength despite being one-half lighter than aluminium makes graphene a highly sought material to be used in any application (Neuberger, Adidharma, & Fan, 2018).
2.3 Rheological behaviour of drilling fluids

Rheology is coined from Greek words ‘rheo’ - flow and ‘logia’- study of; which refers to the study of deformation of matter and flow of fluid under tensions. The rheological behaviour is a function of shear stress (shear force per unit area) and strain rate (change of strain with time) relation. Since viscosity is defined as the ratio of shear stress and strain rate, it is considered as the basic element to ascertain the rheological activity of fluids and measure viscid resistance to flow. The higher the viscosity, the more energy/ power is required to pump the fluid and the higher the pressure drop. Other rheological properties of drilling muds that are necessary to be controlled for an effective drilling management are for instance mud density, gel strength, plastic viscosity (PV), apparent viscosity (AV), yield point (YP), filtrate loss volume and lubricity.

Ahmadi Nadooshan, Eshgarf, & Afrand (2018) noted that the rheological behaviour of nanofluids is significantly affected by many parameters such as base fluid type, nanoparticle type, structure, size and weight percent, temperature, and shear rate. L. Wang, Chen, & Witharana (2013) concluded that the main factors that influence nanofluid rheology are nanoparticles concentration and their Brownian motion.

An ideal drilling mud must be able to suspend drill cuttings at stationary situation whilst simultaneously able to circulate when low force is employed. High enough viscosity enables solid fragments to be suspended while avoiding sagging concurrently. Theoretically maximum 3 vol% nanoparticle loading in a mixture is permissible because greater particle concentration will cause higher pressure drop and cost of pumping operation. The mud also ought to be stable at extreme temperature to avoid overheating of drilling components. Plus, it is supposed to create thin, impermeable mud cake to lessen fluid invasion into the reservoir. The benefits of incorporating graphene include rheology modification, decline of filtration loss, reduction of coefficient of friction (CoF), increase of the heat transfer rate, shale inhibition and well strengthening.
### 2.4 Review of rheological behaviour of graphene in drilling fluids

#### Table 1. Summary of the studies on rheological behaviour of graphene in drilling fluids

| Author | Types of nanoparticle | Base Fluid | Investigated Properties | Experimental Conditions | Summary of Results |
|--------|------------------------|------------|--------------------------|-------------------------|-------------------|
| Nasser (2013) | Nanographite | Oil | - Viscosity | Low Pressure Low Temperature (LPLT) | 40 nm graphite increases viscosity of OBM and reduces volume filtrate loss by 50% under LPLT condition. |
| (Madkour, Fadl, Dardir, & Mekewi, 2016) | 1. Graphene 2. Multi-walled carbon nanotube (MWCNT) | Oil | - HPHT fluid loss | High Pressure High Temperature (HPHT) | 0.5 wt% graphene and MWCNT in an OBM shows mud filtration reduction under HPHT at 302 °F and 500 psi. |
| (Aftab, Ismail, & Ibupoto, 2017) | 1. Nanosilica 2. Multi-walled carbon nanotube (MWCNT) 3. Graphene nanoplatelet (GNP) | Water | - API and HPHT filtration - Plastic viscosity (PV) - Yield point (YP) - Shale inhibition | Ambient conditions and HPHT (filtrate volume) | Graphene nanoplatelet (GNP) better enhances WBM performance of the KCl mud system compared to the other nanoparticles. |
| (Mohd Taha & Lee, 2015) | Graphene | Water | - API fluid loss - Lubricity - Shale inhibition | LPLT and high temperature (120,oCand 351,oF) | Graphene reduces up to 30% API fluid loss of a 10 ppg WBF and improves lubricity of the drilling fluids. |
| (Ho, Yusup, Soon, & Arpin, 2016) | Graphene Nano-Sheets | Hydrogenated Oil | - Viscosity - Shear stress | 30,oC - 50,oC. | Graphene nano-sheets improve drilling bit life span and fluid loss control despite a slight shear thickening of based fluid at higher shear rates due graphene aggregating when it undergoes rheology test. |
| (B. Wang, Wang, Lou, & Hao, 2012) | Graphite | Oil | - Thermal conductivity - Rheological property | 30,oC - 60,oC. | By only adding small percentage of graphite (1.36 vol %), the base oil shows high thermal conductivity of 36%, significant viscosity increase and shear thinning behaviour. |
Graphene pertaining to the petroleum industry is a topic of interest only of late, hence small numbers of literature could be found that reports on its function and properties in drilling muds (Neuberger et al., 2018).

The rheological and filtration properties of graphite, a carbon-based nanoparticle in OBM were investigated by Nasser (2013). They showed by adding 40 nm graphite, the drilling mud viscosity increased while the mud filtrate volume had been cut by half. The viscosity reduces with the increase in temperature and shear rate.

Graphene was tested in WBM by Mohd Taha & Lee (2015), and likewise the drilling fluids displayed an improvement in rheological performance. The concentrations of graphene were varied from 1 - 5 wt% and tested at temperatures up to 350°F, and an API filtrate loss reduction of 30% was achieved. 80% torque was reduced for a 5 wt% sample of graphene at 200°F condition, besides increasing 10% shale recovery. At a field trial in Myanmar, graphene enhanced mud tripled the rate of penetration (ROP), reduced Coefficient of friction (CoF) from 0.21 to 0.08 which is quite similar as synthetic based muds range and also lengthen the drilling bit’s lifetime by more than 75%.

Madkour et al., (2016) carried out an experiment under HPHT condition at 300°F and 500 psi for 0.5 wt% graphene and MWCNT based biodegradable composites in OBM. When compared to the commercial viscosifier, the graphene-added drilling muds demonstrated a substantial increment in PV, AV, YP and gel strengths and stable behaviour at high temperature. The fluid is found to obey the Herschel-Buckley model.

On the whole, carbon based nanoparticle is shown to stabilise the drilling fluids and acts as good modifier of their rheological behaviours at HPHT (Farbod et al., 2015). The presence of commercial nanoparticles in the mud resulted in no spurt loss, signifying that the fluid system will not damage the close by shale (Amanullah, AlArfaj, & Al-abdullatif, 2011). They noted that fluid invasion into the formations is minimised hence, very thin and well dispersed filter cake is produced.

2.5 Graphene generation from industrial waste
Despite its application across diverse industries, the expensive price of graphene presents an extra hindrance for its mass production. Meanwhile, industrial waste generation has become a pressing universal issue, so transforming the unwanted, accumulated waste into advance material is a valuable and innovative act intended to counteract the problem. If graphene can be effectively and frugally prepared from wastes, this will benefit many industries from energy to environment.

Even though few studies have shown that graphene can be synthesised from wastes such as vegetable and animal oil waste, industrial waste (Deng, You, Sahajwalla, & Joshi, 2016), newspaper, charcoal (Akhavan, Bijanzad, & Mirsepah, 2014), biomass, disposable paper cups (Raghavan, Thangavel, & Venugopal, 2017) etc., it still lack research on graphene usage and behavioural modification in drilling fluid. The quality metrics of the resulting graphene is not clearly pondered upon for any specific purpose (Raghavan et al., 2017). Consequently, the performance level of graphene made through irregular manner is inexplicit. Graphene, based on their different sources and methods of production may have their own specific application due to its differing properties and quality. For example graphene synthesised from waste dry cell and waste paper could be employed in electronic (Roy et al., 2016) and supercapacitor applications (Singu, 2017). Thorough analysis of nanoparticle properties will make way for the application of graphene in its intended usage. This study is therefore done to bridge the research gap and investigate behavioural properties of the water based mud (WBM) + graphene extracted from unwanted waste.

3 Methodology
The proposed experiment to formulate the drilling mud and investigate its rheological properties is discussed in this paper. All the laboratorial work is carried out based on the recommended practice API 13B-1 for testing WBM (American Petroleum Institute, 2009).

3.1 Nanofluid preparation
Three types of drilling fluids are prepared: basic WBM, WBM + commercial graphene and WBM + waste graphene from welding industry and their properties are compared. Basic WBM is prepared by adding fresh water, Na₂CO₃, bentonite, flowzan, hydrostar and barite. The composition of the muds and properties of graphene are given in Table 2.

![Table 2. Formulation of drilling muds](image)

| Materials                  | WBM | WBM + commercial graphene | WBM + waste graphene | Mixing time, min |
|----------------------------|-----|----------------------------|----------------------|------------------|
| Fresh water, ml            | 350 | 350                        | 350                  | -                |
| Na₂CO₃, g                  | 0.25| 0.25                       | 0.25                 | 2                |
| Bentonite, g               | 15  | 15                         | 15                   | 5                |
| Flowzan, g                 | 2   | 2                          | 2                    | 5                |
| Hydrostar, g               | 0.4 | 0.4                        | 0.4                  | 15               |
| Barite, g                  | 147 | 147                        | 147                  | 5                |
| Graphene, ppb              | -   | 0.05, 0.1, 0.2             | 0.05, 0.1, 0.2       | 5                |

Graphene concentration in the mud formulation for both commercial and waste based graphene was varied at 0.05, 0.1, 0.2 ppb. The mud is mixed in a mixer to ensure homogeneous colloidal dispersion of graphene nanoparticle in the solution. For each concentration, the mud is tested before and after 16 hours aging (heating) at different temperature of 200°F, 225°F and 250°F while the pressure is fixed at 500 psi. This is to investigate the effect of graphene loading and temperature on the rheological properties of the drilling mud.

3.2 Rheological behaviour testing
The rheological behaviour of the drilling muds is tested such as density, viscosity, mud filtrate volume and lubricity.

3.2.1 Density
The density is determined using a pressurized mud balance instrument which gives a more accurate reading of the mass per volume of a drilling fluid compared to conventional mud balance. Both are similar in operation, the difference being that the mud sample can be placed under pressure to reduce the influence of entrained air and reduced it to a negligible volume, thus delivering density reading under conditions which mimics the downhole more closely.

3.2.2 Viscosity
By using a viscometer, the gel strength, plastic viscosity (PV), apparent viscosity (AV), yield point (YP) could be determined from readings at rotor sleeve velocities of 300 rpm and 600 rpm. The slurry sample
is put in a vessel container and the rotor sleeve is submerged to the scribed line, 2.3” above the sleeve bottom. The viscosity measurement consists of minimum time delay at temperature 30°C. The dial reading is recorded at 300 rpm and 600 rpm as $\Omega_{600}$ and $\Omega_{300}$. The initial gel strength is the maximum value obtained when the sample is left untouched for 10s after being stirred at 600 rpm.

PV, AV and YP are calculated using equation (1-3).
\[
PV = \Omega_{600} - \Omega_{300} \quad (Eqn \ 1),
\]
\[
AV = \frac{\Omega_{600}}{2} \quad (Eqn \ 2),
\]
\[
YP = \Omega_{300} - PV \quad (Eqn \ 3),
\]

3.2.3 API Filtration
API filtration is conducted at 100 psi and ambient condition (low pressure, low temperature) before aging using API filtrate tester. The drilling mud sample is poured into the testing chamber and the volume loss is recorded every minute for 30 minutes. The thickness of the filter cake is also recorded.

3.2.4 HPHT Filtration
HPHT filtrate volume is obtained using HPHT filtrate volume tester and Whatman filter paper at 500 psi with the pressure supplied by nitrogen gas source. Each formulation of drilling fluids is studied at 200 °F, 225 °F and 250 °F to determine the filtration trend as a function of temperature. The volume of the filtrate loss is measured every one minute for half an hour. The mud cell is let to cool for 60 minutes before the pressure inside could be discharged. Then the filter paper is detached, gently rinsed, and placed at ambient temperature for one day to permit the vapourisation of water from the mud cake. The filter cake thickness is determined using a Vernier caliper after it has dried up.

4 Results and discussion
4.1 Rheological properties
Rheological properties such as plastic viscosity (PV), yield point (YP), 10 s and 10 min gel strength, API mud filtrate, and HPHT filtrate volume of the reported drilling muds were determined and reported below.

![Figure 4. Plastic viscosity at different concentration of graphene – (a) before aging (b) after aging](image)

4.1.1 Plastic Viscosity of Water-Based Mud With Graphene Before Aging
Plastic viscosity (PV) can be described as the resistance of drilling fluid to flow. This is caused by mechanical friction as a result of the interaction between solid and liquid part of the drilling mud. PV is greatly dependent on the concentration and bulk volume of solids in the suspending liquid.

Fig. 4 (a) shows the PV for a water-based mud with commercial and waste graphene before aging (atmospheric condition). The PV for basic mud was 28.2. The PV decreases as the concentration of commercial and waste graphene increases. This correlates with the finding reported by (A. R Ismail, M. S. A. Rashid and B Thameem, 2018) for 0-0.04 ppb graphene nanoplatelet (GNP) concentration. The decrease in PV with graphene concentration is probably due to the degree of dispersion of graphene which affects the rheological behavior of the mud. Lower PV means mud is easier to flow and the drilling process will be much more rapid. However PV must be high enough to suspend the cuttings at low shear rate (no circulation). The higher PV will increase resistance in the mud hence more power is needed to pump the mud (Piroozian et al. (2012).

4.1.2 Plastic Viscosity of Water-Based Mud With Graphene After Aging

The PVs of drilling fluid with commercial and waste graphene after aging is shown in Fig. 4 (b). After aging, it is found that the PV increases with the increase in concentration of the solids present in the drilling mud. This is due to the agglomeration of graphene nanoplatelets which results in high friction and oppose the fluid flow. However, as the concentration of commercial and waste graphene is increased to 0.1 ppb and 0.2 ppb, the PV then decreases. Graphene has a large surface area per volume and this plays a role in the behaviour of the nanoparticles with the surrounding water-based matrix.

The effect of temperature on PV can also be observed from Fig. 4 (b). The higher the temperature, the lower the PV. A rise in temperature decreases the cohesive and attraction force between molecules. The molecules gain internal energy which leads to the random movement of particles. Thus it is easier for the drilling fluid to flow.

4.1.3 Yield Point of Water-Based Mud With Graphene Before Aging

Yield point (YP) is important in ascertaining the capability of a drilling mud to carry drilled cuttings out of the well annulus. It is known that a fluid with a high YP has a better lifting capacity. Fig. 5 (a) shows the YPs of basic mud and water-based mud with commercial and waste graphene. The YP decreases

![Figure 5. Yield point at different concentration of graphene – (a) before aging (b) after aging](image-url)
with concentration of graphene. As can be seen, waste graphene also follows the same trend as its commercial counterpart. However from previous literature review, yield point is supposed to increase with increase in graphene concentration. The decrement in this study may be explained due to the improper distribution and mixing of the graphene in the drilling fluid.

4.1.4 Yield Point of Water-Based Mud With Graphene After Aging

The relation of YPs of drilling fluid with commercial and waste graphene after aging is illustrated in Fig. 5 (b). After aging, as the concentration increases, the YP also increases. High YP ensues from flocculation of bentonite clay and high concentration of solid. It is also found that YP increases with the increase in temperature. This is because of thermal induced swelling and bentonite flocculation tendency at high temperature.

![Graph showing yield point of water-based mud with graphene after aging](image)

Figure 6. Gel strength at different concentration of graphene before aging – (a) 10 sec (b) 10 min

![Graph showing gel strength of water-based mud with graphene before aging](image)

Figure 7. Gel strength at different concentration of graphene after aging – (a) 10 sec (b) 10 min

4.1.5 Gel Strength of Water-Based Mud With Graphene Before A ging
Gel strength is expressed as the ability of a drilling mud to suspend cuttings under stagnant circumstances. Fig. 6 show the gel strength at 10 s and 10 min of the water-based mud with graphene. The gel strengths for basic water-based mud are 9.46 lb/100 ft² and 16.84 lb/100 ft² for 10 sec and 10 min respectively. Based on Fig. 6, the gel strength at 10 sec and 10 min increases with concentration at 0.05 ppb graphene and decreases when more graphene is added. This result is due to the buildup of solid in the drilling mud. The gel strength of the mud is in the recommended range as given by Mi-Swaco (2016) which is 8–16 lb/100 ft² for 10 sec gel strength and 16–30 lb/100 ft² for 10 min gel strength. Initially the gel strength should be high enough to hold the drilled cuttings but after 10 min, the gel strength should not be more than double of its original value so that not much pressure is needed to get the fluid moving again.

4.1.6  Gel Strength of Water-Based Mud With Graphene After Aging
In Fig. 7, the results depict the gel strengths after aging for drilling fluid with graphene at 10 sec and 10 min. The 10 sec and 10 min gel strength also portrays the same pattern before aging. A drilling fluid with high gel strength is typically avoided because high pump pressure is required to recirculate the mud in the event of pump shut down during fishing operation.

4.1.7  API Fluid Loss of Water-Based Mud With Graphene Before Aging
According to Okon et al. (2014), the drilling mud’s filtration and wall building properties are necessary to provide an insight on the amount of mud filtrate infiltration into the reservoir formation and the amount of filter cake that will be accumulated inside the wellbore layer. Thus, the heart of this paper focuses on filtration control of drilling mud using graphene. Mi-Swaco (2016) mentioned that the standard range of fluid loss must be lower than 15 ml.
Fig. 8 shows the overall fluid loss of drilling fluid with graphene. The fluid loss of the basic mud is found to be at 8.3 ml. This result is attributed to the weak attractive force among the bentonite's molecules in basic mud is inadequate to form a strong linkage that reduces filtrate loss. The drilling fluid with graphene samples demonstrate small variances in API filtrate loss volume compared to basic mud; 7.6 ml and 7.2 ml for 0.05 ppb commercial and waste graphene respectively. With a rise in the graphene concentration, the fluid loss decreases since there is a crosslinkage between mud particles and bridging occurs faster. The graphene particles minimize the porous and permeable condition of the filter cake by consolidating the force between the particles and helps in loss decline. Spurt loss also decreases with concentration of commercial and waste graphene. This indicates that the filter cake is deposited faster compared to basic mud which has higher spurt loss.

4.1.8 HTHP Fluid Loss of Water-Based Mud With Graphene After Aging
At static high temperature filtration, graphene may also assist in suspending other colloids and prevent it from sagging thus minimising the total filtrate loss. However, to verify this, high temperature filtration is conducted to study the flow of colloids in the drilling mud.

As presented in Figure 8, there is a reduction in the volume of filtrate loss when graphene is added into the drilling mud sample. This is due to the fine size of graphene with enhanced structures of nanoplatelets that provides better filling properties and seals the well’s nanopore throats to prevent water invasion. A bridge may be formed when graphene particles settle against each other in the pore throat. The fine-sized particles may then plug the spaces between the larger particles that are previously deposited on the surface of the filter paper. In addition, the nano-graphene materials consist of large surface area per volume; thus, they are more resistant to pressure, and they can seal pore throats.

However, after 0.01 ppb of graphene is added, the amount of mud loss increases. It is believed after some point, adding the concentrations of nanomaterials any further will no longer decrease the fluid loss suggesting its optimum concentrations at 0.05 ppb. Having excessive nanomaterials may reduce cost effectiveness besides inducing formation damage.

Looking at the trend, fluid loss worsen with rising temperatures. This deterioration becomes more profound especially at HTHP conditions (250 °F). Factors such as thermal stability of the graphene nanomaterial itself may also cause the disparity in fluid loss volume of GNP.

4.1.9 Mud Cake Thickness Water-Based Mud With Graphene After Aging
Filtrate infiltration may lower the reservoir permeability and induce irreversible formation damage; thus, a filter cake must be created as quickly as possible to avoid damage due to solid cuttings. The mud cake thickness is measured to ascertain its capability to decrease filtrate loss. A good mud cake is supposed to be thin and impermeable to ensure the drilling fluid stays in the wellbore. The recommended mud cake thickness from Mi-Swaco (2016) is 1.5 mm for API test.

For every test temperatures, the mud cake thickness of all drilling fluid formulations remains consistent at < 2 mm for API, and <5 mm for HTHP test. The graphene particles succeeds in forming a solid structure of the mud cake. Furthermore, graphene is also able to fill small gaps and provide a better seal. Graphene improves the mud cake thickness of basic water-based mud because small particles are packed firmly in between the large-sized bentonite particles. This proves that graphene is successful in reducing fluid loss.

5 Conclusion
In conclusion, graphene is able to improve the rheological behavior of water-based mud besides reducing the API and HPHT mud filtrate volumes loss. The optimum concentration is taken as 0.05 ppb as it improves the filtrate loss by 8.43% at API condition and 18.57%, 13.82% and 14.06% at 200°F,
225°F and 250°F respectively. Waste graphene follows similar trend as commercial graphene, hence waste graphene is successfully shown to provide a low cost alternative to the commercial graphene. Further research should be done to study the effect of waste graphene on thermophysical properties of water-based mud to ensure the effectiveness of waste graphene. The characterization analysis of commercial and waste graphene should be done to compare between the actual structure and properties of the two since it may influence the behavior of the drilling fluid. Another possible sources of error include the graphene in the mud is not well mixed and inavailability of equipment such as hot rolling oven which can mimic the wellbore condition.

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