

Synthesis of graphene oxide using modified hammers method as fluid loss control additive for water-based drilling fluid (WBDF)

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Abstract

Water-based drilling fluid (WBDF) is one of the commonly used fluids in drilling operations due to its low cost and environmental advantages compared to oil-based fluid (OBF) and synthetic-based fluid (SBF). The effectiveness of a drilling fluid is mainly dependent on its rheological and filtration properties and these properties are strongly correlated to the type of additives used during the formulation of drilling mud. This study aims to investigate the use of graphene oxide (GO) as a nano-additive in WBDF and evaluate its influence on the rheology and filtration properties of the drilling fluid. Basic rheological properties such as plastic viscosity, yield point, filter cake thickness and density were examined. GO was synthesised using modified Hummer's method and characterised using x-ray diffractometer (XRD), Fourier transform infrared spectroscopy (FTIR), thermogravimetric analyser (TGA) and scanning electron microscope (SEM) to confirm the properties of GO. XRD analysis shows that graphite was successfully oxidised and exfoliated to GO. The FTIR analysis confirms the presence of C–O, C=C, and O–H bonds in the synthesised GO. The amount of GO added to the WBDF was varied and changes in the rheological properties (density, plastic viscosity, yield point and mud cake thickness) were studied. It was observed that drilling fluid developed using GO has better rheological and filtration properties than drilling fluid formulated without GO. The mud thickness was reduced after the addition of GO, indicating that GO-WBDFs have the capability to control fluid loss into the formation during drilling activities.

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1.0 Introduction

Drilling fluid, also known as drilling mud, plays a significant role in the success of many drilling activities such as coal mining and hydrocarbon exploitation. Among the essential functions are to carry cuttings from the wellbore up to the surface, prevent formation damage, support the wellbore, as a lubricant to drill pipe, prevent pipe corrosion, and produce a thin-permeability cake that seals permeable formation (Caenn & Chillingar, 1996; Hossain & Apaleke, 2018; Meng et al., 2012). Drilling fluids can be categorised into water (WBDF), oil (OBF), and gas (GBF), which acts as the base fluid for the drilling fluid. It is a complex system comprising three phases namely; (i) continuous, which is the base fluid that makes up most of the drilling fluid volume, (ii) non-reactive, which is made up of inert solids such as drill cuttings, weighting materials and lost circulation materials, and (iii)

reactive, which consists of different types of additives or treating agents that are capable of modifying the final properties of drilling fluid by reacting with the base fluid (Dyke, 2000). WBDFs have been extensively used to drill wells around the world due to their economic and environmental benefits (Baba & Belhadri, 2009). However, they also can dissolve salt particles, resulting in a disruption in density and disintegration of clay especially in shale formation.

Fluid loss happens when drilling fluid flows into the geological formation instead of returning up the annulus. It can occur during static (e.g. tripping) and dynamic (mud is circulating in the wellbore) conditions (Taiwo et al., 2011). The invasion of drilling fluid into the formation will cause formation damage and lead to a serious problem during the drilling of an oil well or a gas well. The solid residue of the drilling fluid will deposit a layer of mud cake on the wall of the borehole

(Arif et al., 2022). The thickness of mud cake is strongly linked to the torque energy needed to rotate the drill pipe, drilling time, and drilling cost (Lyons, 2016). In a high permeability formation, the high-pressure difference caused by the drilling fluid invasion into the formation can cause the drill pipe to get stuck into the thick mud cake. This situation is called differential sticking. To prevent the invasion, fluid loss additives are commonly utilised to reduce drilling fluid loss by forming a protective layer around the wellbore (Retnanto et al., 2023; Sun et al., 2021).

Most of the drilling fluids are formulated based on micro and macro-sized additives such as lignin, asphaltite and bentonite clay (Fink, 2021). However, some of them are still unable to perform the intended tasks due to their poor physical, chemical, mechanical, and thermal properties. Normal fluid loss additives with diameters in the range of 0.1 to 100 μm are not effective in reducing fluid loss in formations with pore size less than 0.1 μm such as shale (Razak Ismail et al., 2014). Both technical and environmental challenges greatly escalate the cost of drilling a well. This has motivated researchers to look towards nanomaterials as the potential candidates that can offer the solution to the issues above due to their superior characteristics such as toughness, lightness, and corrosion resistance, which are among the main elements required in successful drilling operations.

Many researchers have proposed several types of nano-additives such as metal oxides and carbon nanotubes in designing drilling fluid to meet some functional requirements such as the appropriate mud rheology, density, mud activity and fluid loss control properties (Abdo & Haneef, 2012; Taha et al., 2015). All these properties depend on the reservoir condition such as reservoir pressure, temperature, and depth. By using nanoparticles, clay swelling, spurt loss, and mud loss due to circulation can be minimised as a thin layer of non-erodible and impermeable nanoparticle membrane is formed around the wellbore (Kosynkin et al., 2012). Furthermore, the quantity of nanoparticles needed is small and this can significantly reduce the cost of WBDF (Amanullah et al., 2011; Zakaria et al., 2012).

Graphene oxide (GO) is a monolayer of graphite oxide that can be easily prepared using the modified Hummer's method, in which well-exfoliated GO sheets can be formed through sonicating or mechanical stirring (Compton et al., 2010). GO has excellent properties due to the presence of various oxygen-

containing groups (mainly epoxy and hydroxyl groups) in the graphene-based lattice. The GO sheets perform well at substantially lower concentrations in WBDF compared to other types of additives owing to the nano size. GO also has many benefits to offer to the oil and gas industry as it is environmentally friendly and cost-effective. Natural graphite, which is from inexpensive agricultural waste can be used as feedstock for GO production and the production process can be easily scaled up (Somanathan et al., 2015). By understanding the relationship between GO and the rheological properties of WBDF, the material properties can be easily tailored to achieve better performance of drilling fluids. In this study, the use of graphene oxide as a potential additive to improve the rheological and filtration properties of WBDF was investigated.

2.0 Methodology

2.1 Chemicals and materials

Graphite powder used for the synthesis of GO was obtained from COMAK Sodium nitrate (NaNO_3 , SYSTERM), hydrogen peroxide (35% H_2O_2), potassium permanganate (KMnO_4), sulphuric acid (95–98% H_2SO_4) and hydrochloric acid (37% HCl) were obtained from R&M Chemicals. All the chemicals were of analytical grade and used as received without further purification.

2.2 Synthesis of graphene oxide (GO)

The synthesis of GO was carried out by using the modified Hummer's method, involving oxidation and exfoliation of graphite powder as shown in Fig. 1. Five grams of graphite powder, 2.5 g of NaNO_3 and 200 mL of concentrated H_2SO_4 were first mixed in a beaker and stirred for one hour in an ice bath. Then, 30 g of

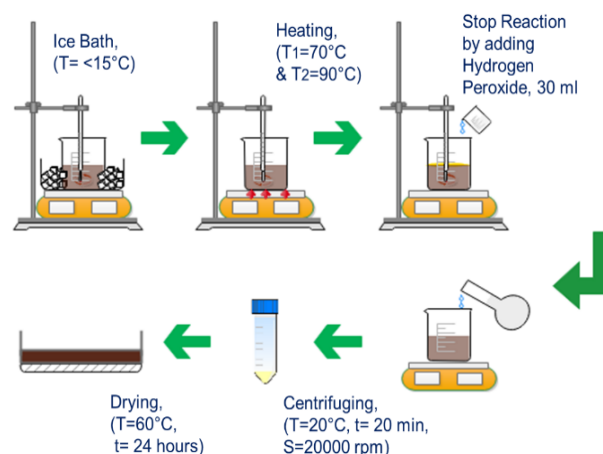


Fig. 1: Schematic diagram depicting the synthesis of GO

KMnO₄ was added gradually over two hours and continuously stirred for another two hours, while maintaining the temperature to be below 15 °C to prevent overheating. Water was added (~ca. 100 mL) to the mixture and the ice bath was then removed to allow the suspension to be at room temperature. The solution was stirred continuously at room temperature for 20 hours. Subsequently, the mixture was heated to 70 °C and stirred for two hours. The solution temperature was then increased to 90 °C and hydrogen peroxide was gradually added to stop the reaction. The resulting mixture was washed repeatedly by centrifugation with HCl and distilled water until a gel-like solution formed and the pH of the mixture become neutral. The gel-like solution was filtered and dried in an oven for 24 hours at 60 °C to obtain GO.

2.3 Preparation of WBDF

Five samples of WBDF were prepared using Hamilton Beach Mixer based on the formula shown in Table 1. The fixed composition for each sample was freshwater (318 g), soda ash (0.15 g), caustic soda (0.2 g), xanthan gum (1 g), barrite (114 g), bentonite (10 g), and hydro-star (3 g). Xanthan is a high molecular weight biopolymer used for increasing the rheological parameters in water-based drilling fluids. Barrite was added to increase the density of drilling fluids, while bentonite, which has a high swelling capacity in freshwater helps to provide viscosity and colloidal solids for filtration control with low clay content. Rheological properties of the WBDF were tested prior to the addition of nanoparticles. The effects of nano additives on the WBDF were investigated by adding different amounts of GO ranging from 3 mg to 12 mg to the WBDF.

Table 1: Formulation of GO-modified WBDF

Sample	GO-0	GO-3	GO-6	GO-9	GO-12
GO (mg)	0	3	6	9	12
Freshwater (ml)			318		
Soda ash (g)			0.15		
Sodium chloride (g)			31.93		
Caustic soda (g)			0.2		
Hydro-zan /xanthan gum(g)			1.0		
Drill-bar/barite (g)			114		
Drill-gel/bentonite (g)			10		
Hydro-star (g)			3		

2.4 Characterizations of GO

The composition and structure of GO synthesised in this work were investigated by using a Rigaku (D/MAX 2200V/PC, Japan) diffractometer with Cu-K α radiation as the X-ray source operated at 40 kV and 40 mA. The diffraction pattern was recorded in the 2 θ range of 5° to 60° with a speed angle of 2°/min. The analysis was performed at a ramp rate of 5 °C/min from room temperature up to 1000 °C in nitrogen environment. Identification and characterisation of GO functional groups were performed on a Perkin Elmer/Spectrum One FTIR spectrometer in the range of 500 to 4000 cm⁻¹. The morphology of synthesised GO was viewed using field-emission scanning electron microscopy (FESEM) (Zeiss Supra 35 VP, Germany).

2.5 Rheological and filtration measurements

Rheological and filtration properties of the WBDF such as density, plastic viscosity (PV), yield point (YP) and mud thickness were measured at room temperature (30 °C). The density test was run using mud balance and the unit is pound per gallon (ppg). The test was carried out according to API 13B standard procedures for testing water-based drilling fluid. The mud balance was set to within 0.5 lb/ft³. The mud cup at one of the beams was balanced by a fixed counterweight at the other end, with a sliding weight along the scale. A level device was placed on the beam to accurately balance the equipment. Meanwhile, plastic viscosity (PV) and yield point (YP) were measured using Fann M3600 Viscometer. The viscometer dial reading was read at 600 rpm and 300 rpm. Each RPM was recorded when the viscometer dial reached a steady reading. The PV and YP were then calculated using below mentioned equation:

$$PV = 600 \text{ rpm reading} - 300 \text{ rpm reading} \quad (1)$$

$$YP = 300 \text{ rpm reading} - PV \quad (2)$$

A standard API filtration test was performed for each sample using an API filter press. The drilling fluid was poured into the cell and a pressure of 100 psi was applied. Filtrate volume was taken at 7.5 and 30 minutes. After 30 minutes, the cell was depressurised and filter paper containing mud cake was removed. The filter cake thickness was then measured using a digital calliper. Each drilling fluid system was examined at least three times to observe any variation and to validate experimental work.

3.0 Results and discussion

3.1 Properties of GO

XRD analysis was used to observe the crystalline structure of the synthesised GO. Fig. 2 compares the XRD pattern and properties of graphite and GO. A sharp diffraction peak was observed for graphite at $2\theta = 26.56^\circ$, which indicates a highly crystalline structure of graphite while the graphene oxide sheet showed a peak at a lower angle; $2\theta = 10.48^\circ$. This proved successful synthesis of GO, which is in good agreement with the literatures (Aba et al., 2015; Paulchamy et al., 2015; Marcano et al., 2010). Meanwhile, the interlayer distance (D-spacing) between consecutive carbon basal planes increased upon oxidation of graphite from 0.34 nm to 0.84 nm, due to the presence of oxygen functional groups and intercalation of water molecules in the carbon layer structure.

FTIR analysis was conducted to confirm the existence of oxygen-containing functional groups, such as C=O and C–O after oxidation. A broad peak at 3243 cm^{-1} was observed, indicating the presence of OH functional group in the GO sheet. Apparent peaks between $1000\text{ to }1800\text{ cm}^{-1}$ indicate carboxyl C=O (1718 cm^{-1}), aromatic C=C (1619 cm^{-1}), and alkoxy C–O (1039 cm^{-1}) bonds. The presence of different types of oxygen functionalities in graphene oxide indicates that graphite has been successfully converted to graphene oxide through modified Hummer's oxidation and exfoliation process. This method is considered a more efficient and less hazardous method for graphite oxidation.

Oxidation of graphite resulted in a brown-coloured viscous slurry, which included graphite oxide and exfoliated sheets along with non-oxidised graphitic particles and residual oxidising agents in the reaction mixture. The GO can be easily distinguished from graphite based on its physical appearance alone. Graphite is grey while GO is blackish brown. Due to the existence of polar groups especially surface hydroxyl groups, GO is highly hydrophilic and contains a large quantity of water, thus a significant increase in mass and its ability to form a gel-like substance can be observed after centrifugation as shown in Fig. 4 (Paulchamy et al., 2015).

Fig. 5 shows the morphology of graphite and the synthesised graphene oxide. The GO had a two-dimensional film-like and multi-layered structure. The

films were stacked above one another and the edges of individual sheets, including kinked and wrinkled areas could be easily distinguished. From the SEM images, the average individual GO sheet was of a thickness between 200 nm to 500 nm. Thicker GO sheets were observed at the edges due to the oxygen-containing functional groups that were mainly combined at the edges of GO (Alam et al., 2017).

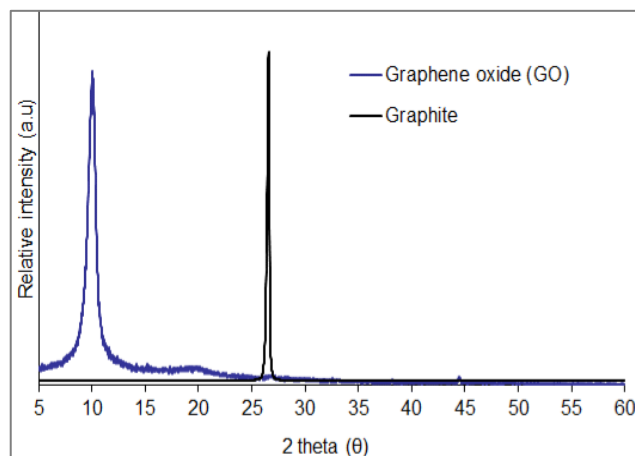


Fig. 2: XRD patterns of graphite and GO

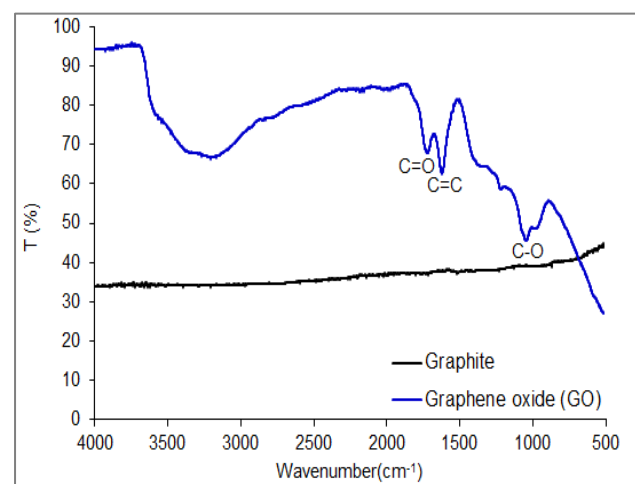


Fig. 3: FTIR of graphite and GO



Fig. 4: Photograph of synthesised GO gel

3.2 Rheological Properties of GO-WBDF

Density is one of the important parameters that need to be considered when developing WBDF. It is used to control the formation pressure and minimise the effects of sloughing shale that might be encountered in stressed areas (Fattah et al., 2011). Therefore, the mud density should suit the condition of the formation during the drilling operation. Table 2 shows the density of WBDF with and without the addition of GO. The value was independent of the amount of GO added as only a slight increase in the density value was observed when GO was added to WBDF as the amount added was very small, between 3 mg to 12 mg.

The rheological behaviour of WBDF is known to be dependent on the particle type, size, amount and interparticle distance of nanoparticles compared to micron-sized and larger particles. Plastic viscosity is the resistance of a fluid to flow and is usually a function of solid additive concentration and shape. If the PV is too high, low rotation per minute of the drill string is usually observed, which can cause several issues such as high equivalent circulation density leading to pipe sticking (Adari et al., 2000). Nanoparticles are known to have a large surface area per volume ratio compared to microparticles. Thus, the interaction between nanoparticles and WBDF is generally higher. Referring to Fig. 6, PV decreased with higher GO concentration. GO is hydrophilic and has a high surface area per volume ratio. This high surface area provides more sites for bonding with functional groups that can influence chain entanglement. Owing to the hydrophilic nature of GO, it could easily form a bond with the matrix of WBDF (water) and lower the WBDF PV. Usually, a high number of nanoparticles in WBDF might increase the PV due to the tendency to form agglomeration and excess colloidal solids. Interestingly, in this experiment, no increase in PV was observed even after 12 mg of GO was used.

YP is the ability of WBDF to move fluid and a measure of attractive forces between drilling fluid particles due to the presence of positive and negative charges (Abduo et al., 2016). It can be seen in Fig. 6 that the YP obtained for WBDF was in the range of YP required (12 to 20 lb/100 ft²). Without any addition of GO, the YP was 22.4 lb/100 ft² and the value decreased to 12.4 lb/100 ft² with the addition of 12 mg of GO. This could be due to GO being easily absorbed onto the surface of the drilling additives' particles, which reduced the agglomeration of the particles.

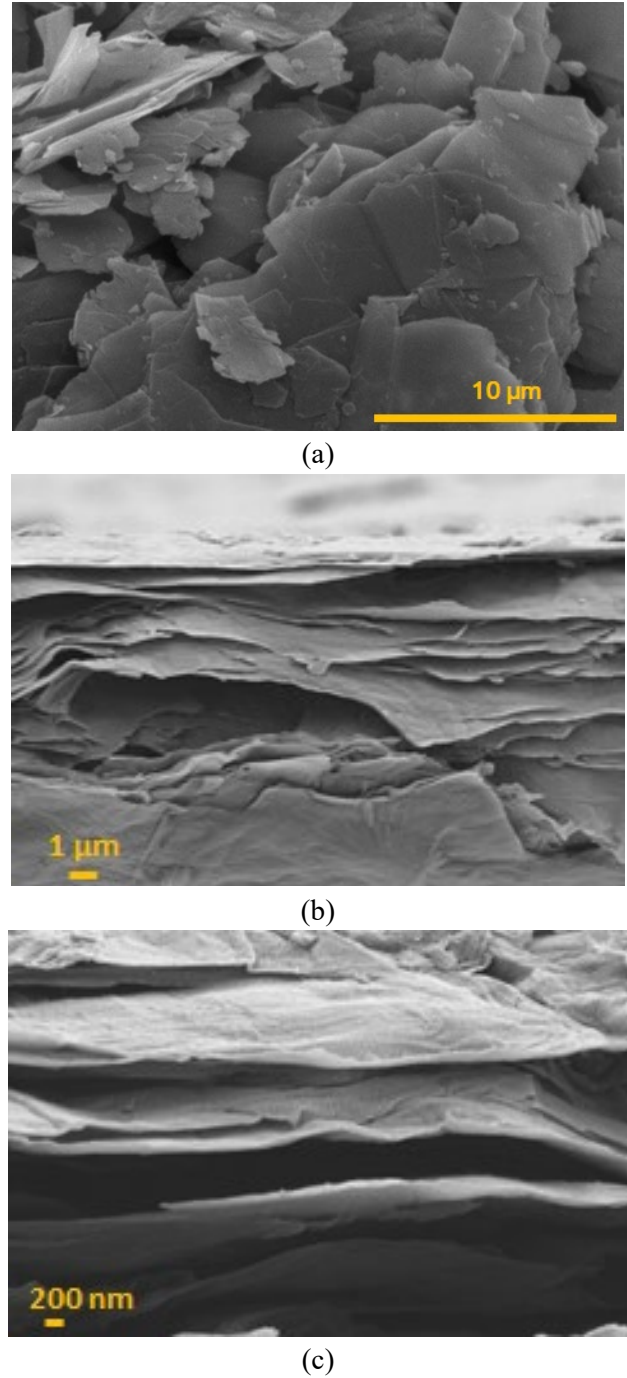


Fig. 5: SEM images of (a) graphite, (b) GO, and (c) GO

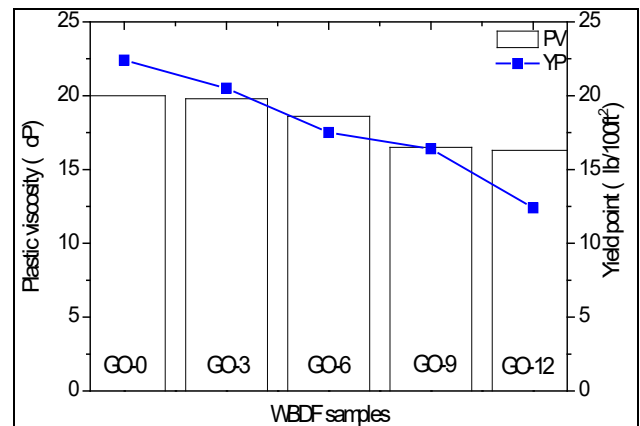


Fig. 6: Plastic viscosity and yield point

Table 2: Density of GO-modified WBDF samples

WBDF samples	Density (ppg)
GO-0	9.6
GO-3	10.25
GO-6	10.45
GO-9	10.65
GO-12	10.6

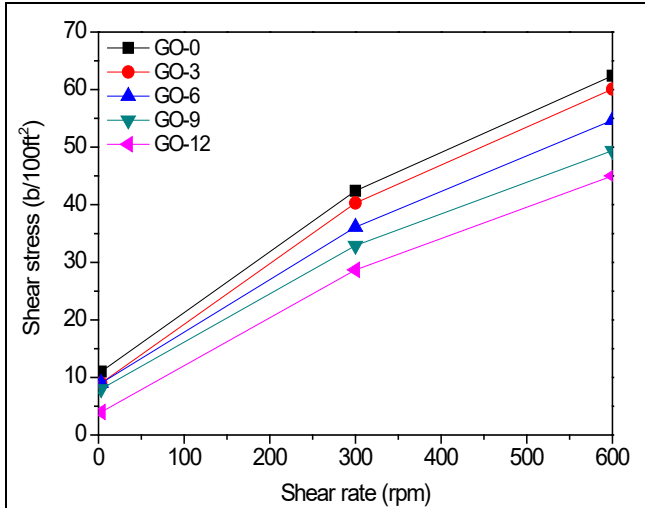


Fig. 7: Flow curve of GO-modified WBDF samples

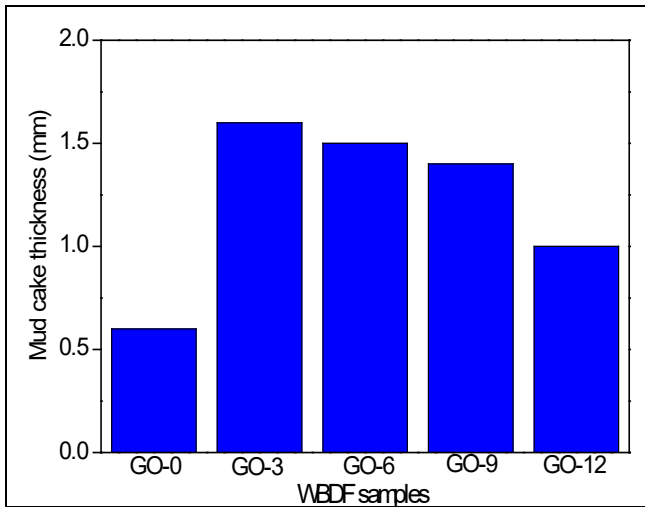


Fig. 8: Mud thickness of GO-modified WBDF samples

Other studies done using bio-based graphene show almost the same result where the PV and YP have a slight increase (Safian et al., 2023). On the other hand, the increase in these two properties was attributed to the overall higher solid content when graphene was added.

Fig. 7 shows that GO modified WBDF prepared in this work was non-Newtonian fluid as the lines were not linear. At low shear rates or velocities, an incremental increase in shear rate produces a high increase in shear stress. However, only a small increase in shear stress was observed while at high shear rates.



Fig. 9: SEM images of WBDF filter cakes without GO (GO-0), with 3 mg (GO-3), and 9 mg of GO (GO-9)

An increase in shear stress can have various effects, either positive or negative, depending on the drilling operation and the characteristics of the drilling fluid. Examples of potential effects are improved hole cleaning, reduced frictional pressure losses and changes in rheological properties.

3.3 Filtration properties of GO-WBDF

Filtration property of WBDF is known to be affected by the amount and properties of colloidal materials used during the formulation. Higher loss of the WBDF can cause invasion of solid particles into the formation, which leads to the formation of internal mud

cake in the wellbore area. This creates a flow barrier and simultaneously reduces the oil and gas flow. By using a sufficient amount of colloidal material, fluid loss can be minimised (Razak Ismail et al., 2014). Fig. 8 illustrates the effects of GO addition on the filter cake thickness of WBDF. The thickness of the filter cake decreased significantly when GO was added to the WBDF. Filter cake is known to be dependent on solids and base water retained in the filter as high solid content usually leads to a thicker filter cake. However, in this work, the thickness of the filter cake decreased with a higher concentration of GO. Among the contributing factors might be the nanometre thickness of hydrophilic GO, which increases the number of active amounts of GO used. This highlights the advantages of using nanoparticles in WBDF formulation, which can reduce flocculation and enhance the suspension of drilling fluid particles resulting in thinner sedimentation and filter cake. Although there is some decrease in mud cake when added GO, the thickest mud cake is without GO. This is because of the solid content of GO causing higher thickness of the mud cake.

Fig. 9 shows the SEM images of the WBDF filter cake without GO (GO-0) and with GO (GO-3 and GO-9) from the top surface. Different forms of microstructures were observed when GO was added to the WBDF. Chunks of big particles disappeared while nanoparticles were immediately visible upon the addition of GO as seen in Fig. 9. As GO was uniformly dispersed in the WBDF, the particles drew closer and their interaction increased, giving a less permeable/compact microstructure. After the addition of 0.9 mg of GO (GO-9), more deformable colloidal particles in the filter cake layer and a reduction in porosity were observed. As a result, a more compact and uniform structure was visible. The same result also shown in other research using synthesised graphene nanoparticle from rice husk char, where the mud cake thickness reduces as the concentration reduce (Yahya et al., 2023). The nanoparticle forms a seal that minimises mud seepage through the retention by passing through

the particles that result lower filtration fluxes and reduce the thickness of the mud cake.

4.0 Conclusions

In this study, graphite has been successfully synthesised into GO by using the modified Hummer's method. The synthesised GO was then used as a nano-additive in the WBDF. The influence of the GO amount added to WBDF on the rheological and filtration properties was investigated. From the results obtained, it can be concluded that the addition of GO leads to WBDF with better PV and YP properties. Furthermore, the filter cake thickness was also observed to decrease significantly with a higher amount of GO. Owing to its nanomaterial properties, the amount required in WBDF is lesser, thus can significantly reduce the cost of drilling fluid. As GO can be easily produced from abundant graphite sources and a common reagent, it can be considered as one of the suitable candidates for the future generation of fluid loss control additive in WBDF.

Contribution statement

Nik Khairul Irfan Nik Ab Lah: Writing original draft and editing; **Wan Al Harrif Alif Wan Zaini:** Writing original draft and experiment; **Nur Hidayati Othman:** Conceptualisation and supervision; **Nur Hashimah Alias:** Data collection and project administration; **Munawar Zaman Shahrudin & Muhamad Shafiq Sayuti:** Data collection and analysis.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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