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Effectiveness of Ultra Sonication on Graphene Characterization in Water Based Drilling Fluid (WBDF) at Elevated Temperature

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Abstract

The main aim of this project is to study the effectiveness of sonication on the graphene characterization in water based mud at elevated temperature. In this project, the graphene sheets will be sonicated using a probe sonicator and added into water based mud. The most optimized sonication time and power that is efficient in giving the best properties of graphene are studied. The sonication time and power must also not damage the graphene sheet properties. In this project study, the graphene sheets are then characterized based on various ways. Sonication of graphene with mud will affect its particle size and number of graphene sheet which will result in change of the drilling fluid properties such as plastic viscosity, yield point and gel strength before and after hot rolled. The properties are studied using equipment such as viscometer, roller oven and mud balance. As for the properties of graphene, it will be separated from the liquid using Low Pressure, Low Temperature (LPLT) filter press equipment and left to dry. It is then examined using FESEM and XRD to give the estimation of spacing and graphene sheet quality.

Keywords: Graphene, Ultrasonication, Sonicator, Nanosheets, Rheology

Introduction

Background of Study

Numerous researchers have conductor an in-depth study by varying the composition of nanoparticles on the enhancement of wellbore stability, filtrate loss and cutting transport ability. However, the research on how these enhancements occur and their underlying changers in physical properties remain unprecedented. Therefore, the gap in this study is to characterize the key mechanisms of particle interactions to further understand the physical interactions between nanoparticles and the common additives present in drilling fluid as recommended [1].

The study on Two-Dimensional (2D) materials has attracted attention worldwide especially after discovering about graphene which happen to be the first 2D material. Graphene is a carbon allotrope made up of carbon atoms arranged in honeycomb-shaped lattice. The carbon atoms in the layers are strongly bonded to 3 adjacent carbon atoms. Hence, the bonds in the layers have an almost perfect strength level. Graphene could be the most promising nanoparticle to be used as an additive in Water Based Drilling Fluid (WBDF) without permanent damage on the formation during drilling. The size of graphene sheet in WBDF can play a vital role to enhance the filtrate loss reduction. The more filtrate loss, the more bore hole stability issues encountered. It will cause the formation of thick mud cake which will eventually reduce the diameter of wellbore causing drill pipe to stuck.

WBDF have been gaining more popularity in worldwide. This trend is influenced due to the changing in environmental regulations and most importantly the advancements in WBDF technology. Adding nanoparticles such as graphene into the WBDF had increase the drilling fluid lubricity and rheology based on recent studies [2]. The ultrasonication could be one of the effective method to modify the graphene characterization by changing the structure of graphene sheet to ensure it is mixed homogenously with the WBDF. Hence, it is important to study the accurate sonication time and power so that the perfect WBDF formulations is formulated. The effectiveness of ultrasonication of graphene is important to make the WBDF a suitable media be used to drill at the rig site later. The role of ultrasonication on graphene structure and characterization with WBDF at elevated temperature is studied in this experimental research project.

Problem Statement

Drilling fluid is one of the important aspects during drilling operations. However, there are several issues with the drilling fluids which includes, pipe stuck, borehole instability, filtrate loss, formation damage, thermal stability and hole cleaning. All of these drilling problems are caused by the degradation of drilling fluids properties such as plastic viscosity, yield point and gel strength. This will cause the increase of filtrate loss which will increase the mud cake thickness. Thick mud cake will result in the diameter of wellbore reducing and get tight. It will cause the drillpipe to get stuck in wellbore. Decreasing gel strength will ultimately reduce borehole cleaning efficiency. As the yield point reduces, it will also cause the mud to loose ability to withstand at high temperature causing the heat at the bottom of formation to not flow to the surface. Currently, materials such as calcium carbonate and nuts are being used in the industry to overcome these issues. Graphene is another alternative to the problems encountered such as filtrate loss, thermal stability and hole cleaning. Graphene sheets can generate smooth film that coats the surface of drill pipe resulting in friction reduction. Graphene also will increase the mud properties such as plastic viscosity, yield point and gel strength by providing more individual particles for the drilling fluid given concentration. However, mixing this graphene with mud is an

elusive process as it cannot be mixed using normal mud mixer. Hence, the use of probe sonicator by carrying out sonication in this mixing process is studied. Sonication of graphene in the mud will affect the properties such as particle size, number of graphene layers, filtration rate which will affect the property and behavior of WBDF in terms of mud weight, plastic viscosity, yield point and gel strength which will be studied in this project [3].

Objectives

The objectives of this study are:

- To investigate the role of ultrasonication on the graphene structure and its characterization at elevated temperature.
- To investigate the role of ultrasonication on the graphene based drilling fluid at elevated temperature.

Scope of Study

In order to achieve the two objectives above, the following scopes will be carried out: The focus for this project includes the role and mechanism of ultrasonication as well as on how this sonication process affects the graphene properties. In this study, the graphene are sonicated before adding into the water based drilling fluid and tested at elevated temperature [4]. The characterization of the graphene is done before and after sonication. The scope of study will be focused on characterization of graphene using Field Emission Scanning Electron Microscope (FESEM) and X-Ray Diffraction (XRD) to study their properties. FESEM is a microscope which works with electrons that liberate by a field emission source. FESEM can be used to visualize very small details on the surface of a nanoparticle such as organelles, polymers and coatings on microchips. This will enable us to look into separated graphene nanosheets properties. On the other hand, XRD is used to study the mineral presence and d spacing exists between the layers of graphene. Higher the d spacing between two layers, bigger the size of material. Hence, we can decide which sonication speed and time was better. Furthermore, to study the ultrasonication of graphene based drilling fluid, the sonicated graphene are added into the mud and mixed using mud mixer as usual. The properties of the mud are then further studied using viscometer and mud balance [5]. Based on the results of the ultrasonication at different time and speed and characterization results from FESEM and XRD, optimum experimental conditions for efficient property of graphene sheet will be determined.

Graphene in Oil and Gas Industry

Oil and gas industry is the biggest sector in the world in terms of dollar value market. This industry is the global powerhouse with thousands of workers worldwide and generating multi-billion dollar globally every year. They contribute a large amount towards national Gross Domestic Products (GDP). New technology had been applying constantly in this industry to make it a sustainable field [6]. The world's fuel consumption is projected to increase in the upcoming years. Hence, it is crucial to apply new technology to this oil and gas industry in line to fulfill the world's energy needs in the future (US Energy Informatin Administration, 2016) [7].

Nanotechnology has shown the potential in oil and gas industry in areas of oil extraction and production. A study by Xuan and Li showed that the use of graphene as additive in mud, reduces the fluid loss level ten times better than bentonite drilling mud. Not only that, graphene are much smaller and denser compared to bentonite. So, graphene can be a viable option for drilling fluid for fluid loss control. Other than fluid loss control, it is also important to look at the rheology of the mud once the graphenes are added [8]. Poor rheology properties will result in low penetration rate causing the drill bit to be hotter. This might cause downtime during drilling. According to a study, it is proved that the use of nanoparticle such as graphene in water-based mud enhances the viscosity of the mud, increasing the mud's gel strength and circulating capacity [9].

In oil and gas drilling applications, studies have proved graphene as promising additives with many benefits. The viscosity of the mud can increase by 16% by adding graphene into it, while behaving similar to Newtonian fluid with Zero shear stress. Adding graphene into mud also reduces the Coefficient of Friction (COF). Reducing the COF will reduce the drag. It will result in better transfer efficiency of energy to the bit. Below we can see the table of the COF when graphene is added to distilled water as comparison (Figure 1).

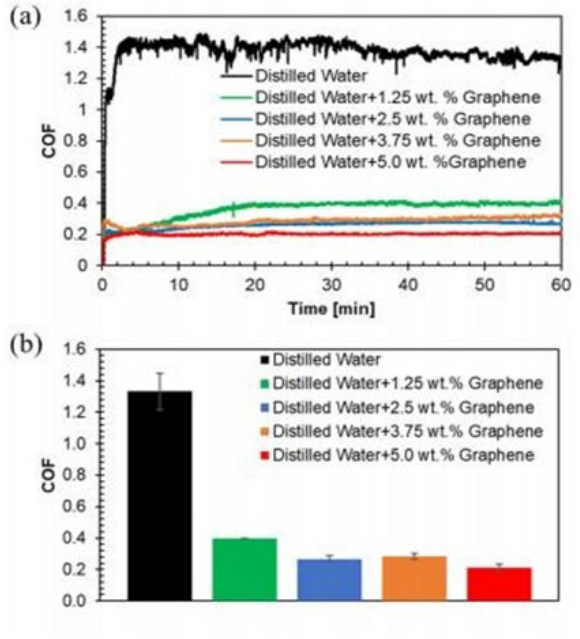


Figure 1: Comparison table between distilled water and distilled water with graphene (a) line graph of COF comparison (b) bar chart of COF comparison.

Ultrasonication of Graphene

Graphene is referring to an atomic layer of graphite in general. In this project, we will focus on conditions to increase the number of graphene sheets as it is almost impossible to produce a single sheet of graphene [10].

The role of ultrasonication in this process will be studied. The ultrasonication method uses a probe sonicator to disperse the graphene sheet and to increase the quantity of it.

The graphene sheets undergo liquid exfoliation and increase the number of sheets. Many studies on liquid exfoliation in order to increase graphene dispersion have been carried out due to its scalability, quality of graphene being produced and simplicity (Figure 2) [11].

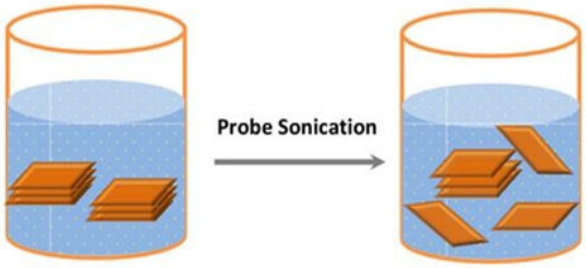


Figure 2: Effects of ultrasonication using probe sonicator to the graphene sheets.

Hence, the probe sonicator will be used to disperse the graphene sheets through liquid phase exfoliation. This method is based on separation of graphene layers with a suitable liquid media [12]. Few graphene layers will be stacked on top of each other. In between these layers, there are weak π-π bonds. This is the bond that needed to be break to disperse the layers. External force is needed to penetrate in between the weak bonds which are achieved through sonication. The choice of liquid media used during sonication also plays a vital role in breaking the weak bonds. The solvent need to have a good surface energy to overcome Van der Waals force in between the graphene and break in between the bond [13].

Hence, the right choice of solvent is important as it determined the strength of the solute-solvent interactions. However, there are drawbacks in the currently used methods which use surfactant to reduce surface energy between water and disperse the graphene sheets [14]. Involving the use of surfactant uses higher cost and it might also be harmful and polluting. A recent review emphasizes the need to sonicate graphene using green and non-toxic dispersants. Looking into this aspect, water will be an ideal solution, as an environment-friendly and low-cost dispersion medium considering graphene is proved to be hydrophilic [15].

Characterization by FESEM and XRD

After sonication, the graphene samples are then kept for agglomeration observation. It is then separated using LPLT and sent to characterize the properties. Sonication process is carried out at different time period and speed to obtain the most optimum condition. We will be able to determine the optimum condition by characterizing the graphene with certain equipment. Properties of graphenes are usually characterized using XRD and FESEM [16]. Both of this equipment is very common for graphene testing. FESEM image shows the agglomerated particles and its morphology. By using XRD data pattern, we can look into the synthesized properties and consolidated samples [17].

FESEM have the ability to examine a very minor area virtually with unlimited depth of field. A field emission cathode in electron gun of a microscope gives narrower probing beams at high and low electron energy. This results in improved spatial resolution and minimizes the damage to the samples. FESEM measurements are carried out to study on the surface morphology and topography of the graphene. FESEM imaging can be taken at different levels of magnification and will be further compared to the atomic force microscope image. FESEM is very suitable for observing small structures in nanomaterial which includes graphene [18]. We can extract data such as quality of graphene sheet and its morphology by using FESEM.

XRD is used to determine the crystallographic structure of a material. XRD works by irradiating the material tested with incident X-rays. It then continues working by measuring the intensities and scattering angles of the X-rays which leaves the material d-spacing in XRD is known as the spacing between the diffracting planes [19]. In the case of graphene, d-spacing helps to determine the amount of graphene sheet it has before and after sonication. XRD is also able to identify structural properties such as grain size, lattice parameters and strain. Hence, XRD analysis will be one of the crucial methods in analyzing the properties of graphene before and after sonication.

Methodology

Approach to Methodology

This chapter will focus on the methodology and experimental work for the project (Figure 3).

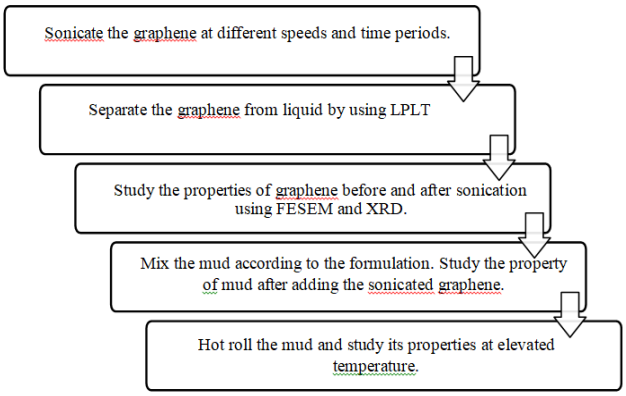


Figure 3: Project methodology layout.

Sonicate at different speed (amplitude) and time

For the first stage of this study, the graphene will be sonicated at different sonication speed at a constant time period. Sonication speed of 25%, 50% and 75% are used at a constant time of 60 minutes. Sonication is done with distilled water of 350mL and graphene quantity of 0.1 g. Before sonicating the samples, it is first mixed using magnetic stirrer to make sure the graphene particles are distributed among the liquid for 5 minutes. And then it is transferred to the sonicator. The beaker with liquid of graphene sample is kept in a container filled with ice cubes.

This is done to maintain the pressure of the liquid while sonication and to prevent overheating of the probe. Next, the probe is lowered till it is covered by the liquid and the time and speed of sonicator is set. The sonication process starts. It is important to observe the equipment and sample every 10 minutes to make sure the ice is not entirely melt. In the case if the ice melts, the sonication process has to be stopped to replace the ice cubes before it is continued again [20]. The process is then repeated with same sonication speed but different time period. The graphene samples are kept constant with speed of 50% with time period varying between 60 minutes, 150 minutes and 240 minutes. After sonication is done the samples are then transferred into glass bottles from the beaker for storage purpose (Figure 4), (Figure 5), (Figure 6), (Figure 7).

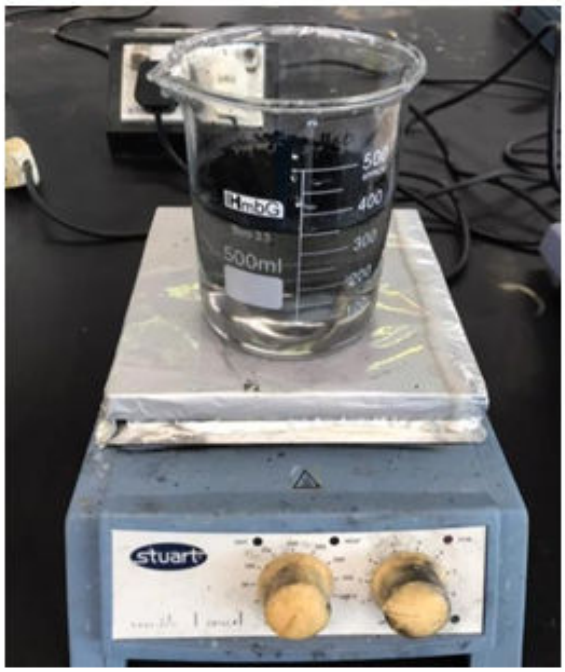


Figure 4: Mixing graphene with distilled water using magnetic stirrer.

Figure 5: Place beaker into container filled with ice.

Figure 6: Sonicator setup.

Figure 7: Set the time and speed of sonication.

Carrying out LPLT filter press to separate the grapheme

After sonication process, the graphene must be separated from the liquid in order to be sending for characterization. This process is done

by using LPLT filter press. The cell must first be assembled starting from the base cap, rubber gasket, screen, filter paper, rubber gasket and then the cell. The liquid can be poured in after the cell and then continue assembling with rubber gasket and finally the top cap [21]. It is placed on the LPLT frame can closed. A beaker is placed below the set up to collect the liquid which will flow out from the cell. Pressure of 100psi is applied till all the liquid flows out. The filter paper is then removed from the cell and left to dry. This process is repeated for all the samples (Figure 8), (Figure 9) [22].

Figure 8: LPLT filter press setup.

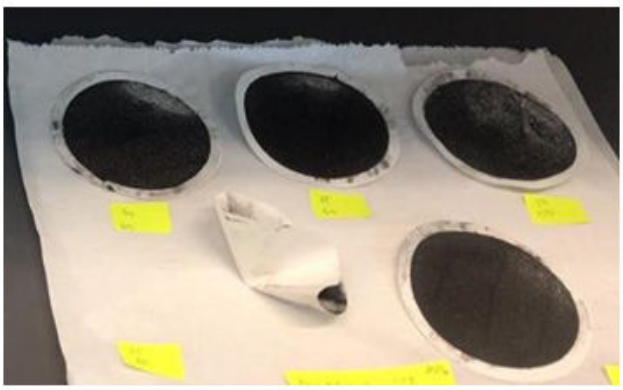


Figure 9: Graphene separated from liquid left to dry.

Characterization using FESEM and XRD

The graphene samples of different speed and time are then characterized using FESEM and XRD. FESEM can be used to visualize very small details on the surface of a nanoparticle will enable us to look into separated graphene nanosheets properties [23]. On the other hand, XRD is used to study the mineral presence and d spacing exists between the layers of graphene. Higher the d spacing between two layers, bigger the size of graphene particle. Hence, we can decide which sonication speed and time was the best [24].

Formulation of WBDF

To study the efficiency of this graphene in real situation, we will need to mix it with mud and analyse the performance. Usually water-based mud is made up from water, potassium chloride, caustic soda, xanthan gum, calcium carbonate, filtrate control agent and barite [25]. The amount of each material is calculated based on its density and the concentration of mud required. In this project, the mud formulation will have extra element which is graphene. The mud mixing is done using normal mud mixer (Figure 10), (Figure 11) [26].

Base Sample

	Concentration	Mixing Order	Mixing Time
Fresh Water	332mL	1	
Soda ash	0.25g	2	2
Bentonite	15g	3	5
PAC-RV	0.3g	4	5
Starch	1.5g	5	5
Caustic soda	0.1g	6	5
Barite	27.99g	7	30

NP Concentration

NP	concentration	Mixing Order	Mixing Time	Sonicated ?
Graphene	0.1 - 0.6 gm	8	60	Yes
hBN	0.1-0.6 gm	8	60	Yes

Figure 10: Mud formulation with graphene for this project.

Figure 11: Mud mixer setup.

Mud balance and viscometer

Before hot rolling the mud, we have to check the mud weight using mud balance. The mud is poured into the cup and closed with a lid. The mud has to overflow from the cup to indicate it is entirely full. The mud balance is then placed on the knife edge in the case. We have to move the rider till the bubble in the level glass is in the middle. The reading is recorded based on the rider position when the bubble is at middle [27].

The next process is rheology reading before hot roll. The rheology reading is taken at 600 RPM, 300 RPM, 200 RPM, 100 RPM, 6 RPM and 3 RPM with viscometer. These readings are used to calculate the plastic viscosity and yield point. Plastic viscosity indicates the solid distribution in the mud while yield point indicates the resistance of fluid to start moving. Plastic viscosity is obtained by minus reading at 600RPM with 300RPM. Yield point value is obtained by minus reading at 300RPM with plastic viscosity reading [28]. We can also obtain the gel strength reading at 10 seconds and 10 minutes by using the viscometer. The mud must be mixed with 600RPM for 10 seconds and turned off. For 10 seconds reading, we need to wait 10 seconds before turning on and get the reading at 3RPM. The same step is repeated for 10 minutes reading. Gel strength indicates the strength of fluid to hold solid. Rheology readings must be taken before and after hot roll the mud to study the changes on the behavior of the mud similar to the mud behavior while drilling (Figure 12), (Figure 13), (Figure 14), (Figure 15).

Figure 12: Mud balance setup.

Figure 13: Reading taken when bubble in the level glass is in the middle.

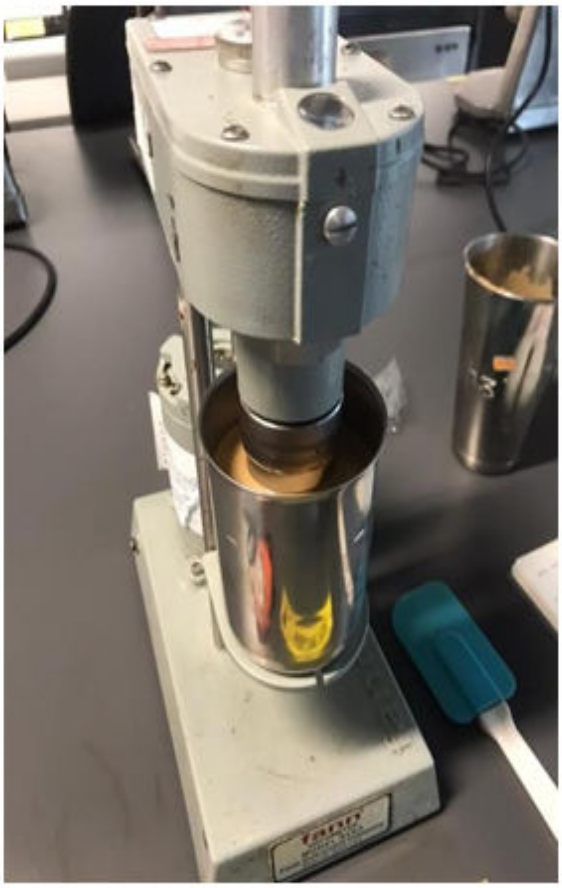


Figure 14: Viscometer setup.

Figure 15: Speed selection for mud rheology.

Figure 16: Pour mud into aging cells.

Figure 17: Aging cells setup.

Hot roll mud using roller oven

In order to imitate the real situation during drilling, the mud is hot rolled using roller oven.

The mud is poured into aging cell and hot rolled at temperature of 150°C and pressure of 100 psi for 16 hours to simulate drilling conditions at rig with water based mud [29].

Before keeping the aging cell in the roller oven, it has to be pressurized first.

After hot roll for 16 hours, the aging cell has to be left cooled before checking the rheology again.

Suitable gloves need to be used while carrying the aging cell to withstand the temperature and the weight (Figure 16), (Figure 17), (Figure 18), (Figure 19), (Figure 20) [30].

Figure 18: Pressurize the aging cell with nitrogen gas.

Figure 19: Set temperature and pressure for roller oven.

Figure 20: Safety gloves to handle aging cell.

Result and Discussion

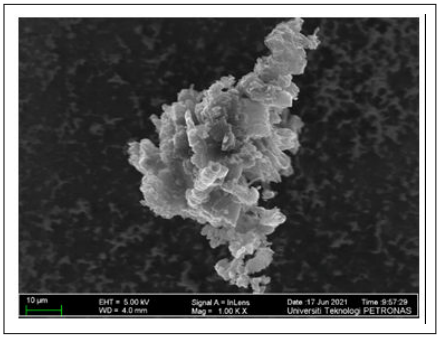
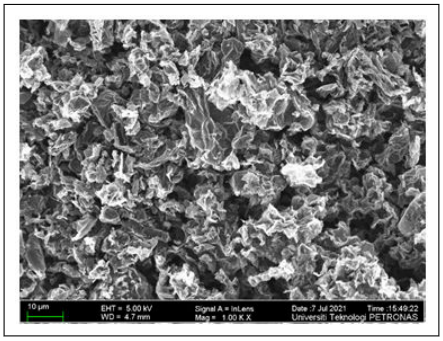
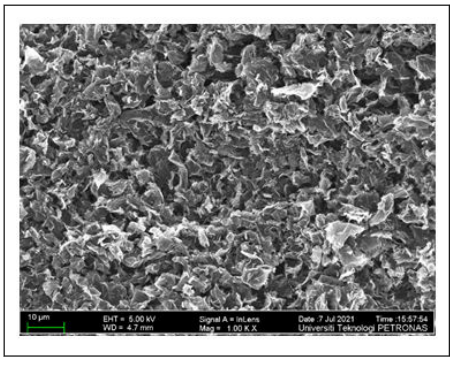
Characterization of graphene

Field emission scanning electron microscope: By using Field Emission Scanning Electron Microscope (FESEM) for this project, the morphology and particle dispersion can be determined.

Hindering of anatase particles can cause the suppression of formation of rutile phase.

The images below show the dispersion of the layers which are being disintegrated eventually [31].

We can also identify that the graphene layers are dispersed after sonication processed was carried out (Table 1).

Sonication properties	FESEM imaging	Evaluation
		No sonication (initial)
Sample 1 time:60mins speed:25%		The graphene are dispersed compared to the initial condition.
Sample 2 time:60mins speed:50%		The graphene layers are smaller as the speed increases. This shows that the graphene particle is decreasing.

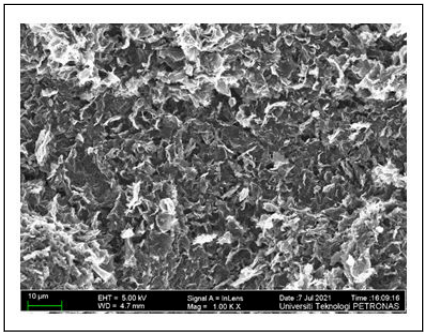
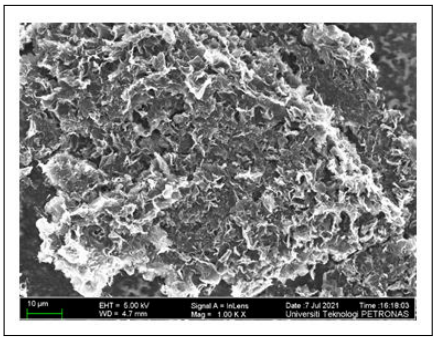
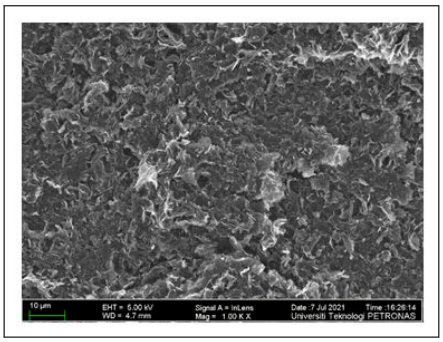
Sample 3 time:60mins speed:75%		Graphene layers are dispersed more. This indicates that the particle sizes are further decreasing.
Sample 4 time:150mins speed:50%		We can determine that the graphene layers start to disintegrate by observing the image.
Sample 5 time:240 mins speed:50%		The layers are totally disintegrated.

Table 1: FESEM images of graphene and evaluation at 1.0 kX magnification.

The percentage weight of the carbon and oxygen from the graphene determined by EDS is shown in table below. The elemental mapping via EDS analysis is carried out to determine the whether the dispersion of these elements in graphene are influenced by the sonication (Table 2) [32].

	Weight		**Atomic**	
	Carbon %	**Oxygen** %	**Carbon** %	**Oxygen** %
Graphene	75.24	24.76	80.19	19.81

Sample 1	77.69	20.56	82.88	16.47
Sample 2	78.19	20.44	83.16	16.32
Sample 3	77.15	20.85	82.59	16.76
Sample 4	77.54	21.73	82.4	17.34
Sample 5	76.97	21.56	82.25	17.3

Table 2: Elemental composition result of EDS of carbon and oxygen element of Graphene.

X-Ray Diffraction (XRD)

The composition, crystallite size and d-spacing of the graphene are characterized by using X-ray diffraction measurements at room temperature. The XRD pattern was collected in the range of 0-90° X-Ray diffractometer. As the d-spacing of the graphene sample increases, it indicates that the particle size is also increasing. When there is presence of sharper and narrower peaks of XRD patterns, it indicates the crystallinity also increases [33]. Based on the results, the d-spacing between tall the samples were very little and almost the same. There was slight decrease in the d-spacing with increasing speed and increasing time (Table 3).

Sample	FWHM	Crystallite size (nm)	2θ position	d-spacing
Graphene base	---	---	---	---
Sample 1	0.921	9.19	22.6395	0.392767
Sample 2	0.8699	9.74	22.8147	0.38979
Sample 3	0.2558	33.12	22.9685	0.387214
Sample 4	0.2303	36.78	22.8175	0.389743
Sample 5	0.307	27.59	22.6579	0.392451

Table 3: Structural properties of the graphene samples.

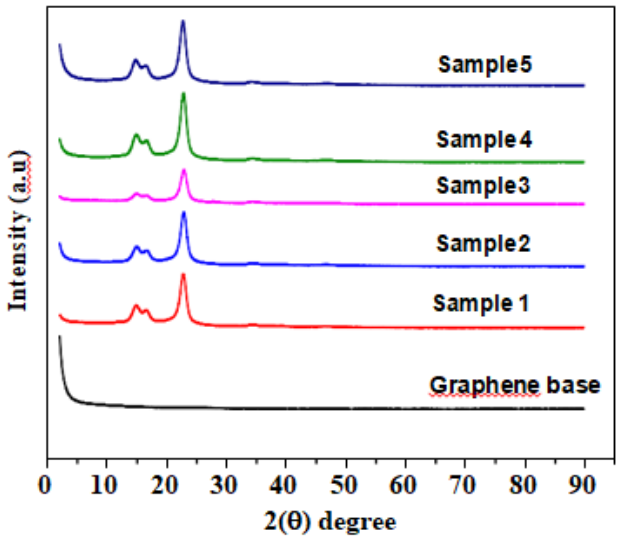


Figure 21: XRD patterns for the graphene samples.

Graphene in water based mud testing

The sonicated graphene are further tested by adding into formulation of water based mud. This is carried out to investigate the role of sonicated graphene on water based drilling fluid at elevated temperature. The water based mud is formulated based on the formulation prepared. Mud weight of each of the mud is recorded. For all the samples, the mud weight did not very much. This indicates that the sonication parameters and graphene do not affect the mud weight [34].

The mud is then transferred to viscometer to check the rheology. Mud rheology is the flow behavior of the mud, which is performed to observe the behavior before and after hot roll. The rheology readings for the muds with graphene are higher compared to the base mud. For the base mud the readings increase after hot roll [35]. As the speed of sonication for the graphene increases, the readings after hot roll decrease but work otherwise for the time change. Comparing the entire after hot roll readings, there is a clear increasing trend as the speed and time period increases (Figure 22), (Figure 23) [36].

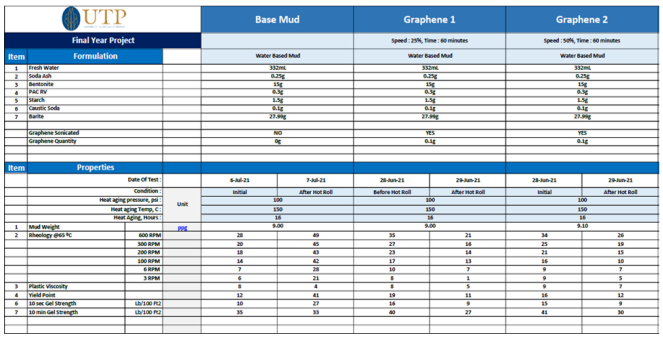
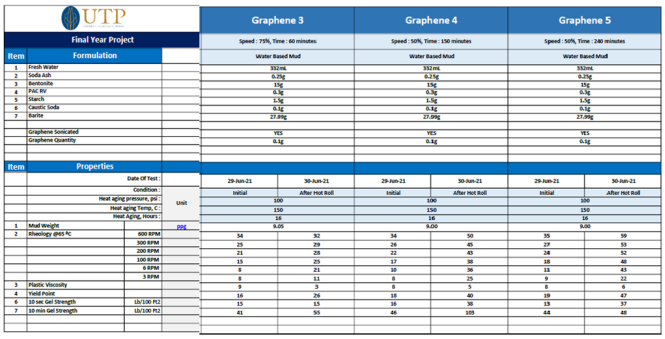


Figure 22: Properties of WBM.



UTP	Graphene 3	Graphene 4	Graphene 5
Final Year Project	Speed : 75%, Time : 60 minutes	Speed : 50%, Time : 150 minutes	Speed : 50%, Time : 240 minutes
Item Formulation	Water Based Mud	Water Based Mud	Water Based Mud
1 Fresh Water	332ml	332ml	332ml
2 Soda Ash	0.25g	0.25g	0.25g
3 Rentonite	15g	15g	15g
4 PAC RV	0.5g	0.5g	0.5g
5 Starch	1.5g	1.5g	1.5g
6 Caustic Soda	0.1g	0.1g	0.1g
7 Barite	27.99g	27.99g	27.99g
Graphene Sonicated	YES	YES	YES
Graphene Quantity	0.1g	0.1g	0.1g
Item Properties			
Date Of Test	29-Jun-21 / 30-Jun-21	29-Jun-21 / 30-Jun-21	29-Jun-21 / 30-Jun-21
Condition	Initial / After Hot Roll	Initial / After Hot Roll	Initial / After Hot Roll
Heat aging pressure, psi	100	100	100
Heat aging Temp, C	150	150	150
Heat Aging, Hours	16	16	16
1 Mud Weight ppg	9.05	9.00	9.00
2 Rheology @65 ºC 600 RPM	34 / 32	34 / 50	35 / 59
300 RPM	25 / 29	26 / 45	27 / 53
200 RPM	21 / 28	22 / 43	24 / 52
100 RPM	15 / 25	17 / 38	18 / 48
6 RPM	8 / 21	10 / 36	11 / 43
3 RPM	6 / 11	8 / 25	9 / 22
3 Plastic Viscosity	9 / 3	8 / 5	8 / 6
4 Yield Point	16 / 26	18 / 40	19 / 47
6 10 sec Gel Strength lb/100 Ft2	15 / 15	16 / 38	13 / 37
7 10 min Gel Strength lb/100 Ft2	41 / 55	46 / 103	44 / 48

Figure 23: Properties of WBM.

Conclusions

The sonication effects on graphene and role of sonicated graphene in water based mud at elevated temperature were successfully investigated. Comparing all the samples, sample 3 gives the best readings. This graphene was sonicated at speed of 75% for 60 minutes. At this point, the graphene layers were expanding but did not disintegrate from the graphene structure. We can clearly see the graphene particle size is increasing by referring to the plastic viscosity. As the time increased to 150 minutes, we can see the sudden drop of plastic viscosity which indicates that the graphene layer have disintegrated from the structure causing the size to reduce. The mud rheology for this sample shows some increase after hot roll. Yield point for the Sample 3 gives the most optimum value which is between 20 to 30. If the yield point increases, higher the resistance of the fluid to start to move which is not preferred. Optimum values are reasonable for the conventional wells. Lower plastic viscosity is preferred for the mud. Lower the plastic viscosity, better the rheological properties for the drilling fluid in terms of drilling operations as it indicates the distribution of solid in the mud.

For recommendations, surfactant can be added during the sonication method for a better separation of graphene layers. Other mud tests, such as HPHT or Permeability Plugging Apparatus (PPA) can be carried out for the muds to test its ability as filtration agent and lost circulation material. From the FESEM results, there were presence of some extra elements which are silicon and iron, which gives a total of about 1% of the entire element. This might be caused due to contamination on the glassware. Hence, glassware needs to be cleaned more effectively using ultrasonic cleaner to prevent this issue.

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