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The Effect of Graphene Oxide Biomass as Filtration Loss Control Agent of Water-Based Mud Fluid

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Abstrak

Lumpur pemboran berbasis air adalah sistem kimia kompleks yang penting untuk penggalian pemboran. Biomassa Graphene Oxide (GO) merupakan material yang memiliki ketebalan submikrometer dengan sifat yang unik dan spesifik. GO dapat sepenuhnya kedap terhadap cairan, uap, dan gas. GO memiliki sifat dispersibilitas terhadap air dan luas permukaan spesifik yang besar sehingga menjadi material potensial yang cocok untuk menjadi agen pengontrol filtration loss pada lumpur pemboran. Tujuan dari penelitian ini adalah untuk menganalisis efektivitas biomasssa Graphene Oxide Cangkang Kelapa Sawit sebagai material additif untuk mengontrol kehilangan cairan pada saat dilakukan proses pemboran. Pembuatan sampel biomassa GO dimulai dari Cangkang Kelapa Sawit menjadi biomassa grafit, kemudian biomassa grafit disentesis menggunakan metode ultrasound-assisted Liquid Phase Exfoliation (LPE) untuk mendapatkan biomassa GO yang tipis. Biomassa Grafit dan Biomassa GO dikarakterisasi menggunakan FTIR, Spektroskopi UV-Vis dan SEM-EDX. Sampel fluida pemboran dengan additif biomass grafit dan biomass GO disediakan dengan penambahan masing-masing 0,5 gr biomass grafit dan biomass GO ke dalam fluida dasar (fluida lumpur bentonite berbasis air). Filtration loss dan ketebalan mud cake sampel diukur dan dibandingkan. Berdasarkan hasil pengukuran, additif biomass GO sangat efektif mengurangi kehilangan fluida berbanding additif biomass grafit. Volume fluida yang hilang berkurang dari 13,9 ml menjadi 10,8 ml berbanding biomass grafit hanya mampu menurunkan menjadi 12,3 ml.

Kata kunci: Graphene Oxide, Grafit, *Kehilangan Filtrat, Lumpur Pemboran*

Abstract

Water-based drilling fluids are complex chemical systems that are essential for water drilling excavation. Biomass of Graphene Oxide (GO) is a submicrometer-thick material with unique and specific properties. GO can be entirely impermeable to liquids, vapors, and gases. It has water dispersibility and a huge specific surface area that is the potential material suitable for filtration loss control agents of water-based mud fluid. This study aims to analyze the effectiveness of Oil Palm Shell Graphene Oxide Biomass as an additif material to filtration control in water-based mud fluid. Making GO biomass sample starts from biomass Oil Palm Shell into graphite biomass, then the graphite biomass was sinthesized using an ultrasound-assisted liquid Phase Exfoliation (LPE) method to obtain thin GO biomass. Graphite biomass and GO biomass were characterized using FTIR, UV-Vis Spectroscopy and SEM-EDX. Drilling fluid samples with graphite biomass and GO biomass were prepared by adding 0.5 gr Graphite biomass and GO biomass into the based fluid (water-based bentonite fluid), The Filtration loss and the mud cake thickness of all the samples are measured and compared. Based on the measurement results, GO biomass additive is very effective in reducing fluid loss compare to graphite biomass additif. The volume of fluid lost was decreases from 13.9 ml to 10.8 ml compare to graphite biomass which was only able to decrease it to 12,3 ml.

Keywords: Graphene Oxide, Graphite, Filtration Loss, Drilling Fluid

1. INTRODUCTION

The fluid loss is one of the main concerns in drilling process in the oil and gas industry. The fluid loss is the loss of volume of drilling mud filtrate during the drilling process due to the mud passing the formation through the formed filter cake (Khodja et al., 2010). It occurs when the formation pressure is lower than the hydrostatic pressure (Ghazali et al., 2015). Filtration loss may generate several problems in drilling operations, such as formation damage if it occurred in the productive zone, blowout, stuck pipe and no drilling

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cuttings were found as log samples. One of the solutions to minimize fluid loss is by adding conventional Lost circulation materials (LCMs) additives into the fluid. Certain conventional LCMs include *sodium bentonite*, *pregelatinized*, *carboxymethylcellulose*, *lignite*, *polyacrylate*, *polymers* (Hamida et al., 2010), *silica* (Medhi et al., n.d.; Parizad et al., 2018), and *calcium carbonate* (Dehghani et al., 2019). However, the deformability and expansion of the conventional LCMs are poor, which makes it difficult for LCMs to enter the lost formation (Song et al., 2019). LCMs offer limited features which increase drilling operation time are and too costly (Zamani et al., 2019) (Ghazali et al., 2014).

The drilling fluid must have little or no aromatic compounds. Also, it is necessary that the drilling be naturally degradable and not have a health impact on workers and the community around the drilling location (Adesina et al., 2012) (Behnamanhar et al., 2014). It is important to find alternative materials that can be used as additives, reduce cost, and provide environment friendly. One of the materials that has a big opportunity as an alternative to additive drilling fluid currently is nanomaterial. Nanomaterial is atomic or molecular engineered material in nanometer scale, usually range from one to one hundred nanometer. The characteristics of the single atoms and molecules differ significantly from those of larger particle. The fascinating quantum and topological phenomena that matter shows at the nanoscale are the sources of nanotechnology's interest. In reason years, numerous researchers have been published on the formulation of drilling fluid using nanomaterials as an additive. These drilling fluids are called nanofluids and they can be defined as any fluids containing at least one additive having a particle size of less in the range of 1–100 nm (El-Diasty and Ragab, 2013). Silica nanoparticles are one of the most commonly used additives to form nanofluids due to their ability to reduce water invasion in shale and control rheology and filtration (Fakoya and Shah, 2017). They have proven themselves in water-based muds, especially KCL muds, significantly reduced fluid loss and enhanced the rheology.

Another nanomaterial development that is of great interest now is 2-dimensional graphene oxide nanosheets. GO is a derivative of graphene which was discovered in 2004 and is considered the most phenomenal nanomaterial due to the lightest and strongest material (Novoselov et al 2004). GO have layered carbon structure bonded to each other to form hexagonal rings. In the structure of a single layer of graphene oxide there is a bend due to the presence of oxygen groups in the form of carboxyl and carbonyl in it (Smith et al., 2019). Scanning tunnelling microscopy shows that the form of oxygen is bound in a rectangular pattern with a lattice constant of 0.27 nm x 0.41 nm. It has a small pore size, large surface area and interesting optical and electronic properties (Dreyer et al., 2010)(Nanda et al., 2015). Graphene Oxide is a strong material that is more water-resistant, has *hydrophilic oxygen*-containing functional groups on a surface (Fu et al., 2020), and has high thermal conductivity, thus improving the thermal stability of the drilling fluid (Aramendiz & Imqam, 2019). GO upon oxidation is *hydrophilic* and water-soluble (Banerjee, 2018). The hexagonal pattern of GO helps bind fluid into the formation. These can be beneficial properties of making materials to prevent filtration loss in the drilling process. Other than that, GO offers a prospective technology that is environmentally- and economical-friendly (Kosynkin et al., 2012).

GO is typically synthesized from natural graphite. Graphite is a naturally occurring mineral that can be found in metamorphic rock, and it can also be synthesized silicon carbide (SiC) through various methods. In this study, researchers are interested by utilizing oil palm shell biomass as a base material for graphite and exfoliated to obtain GO biomass. Because GO from the plantation and agricultural waste for the petroleum industry has not been well-developed. It is a big challenge to use biomass, particularly in Indonesia, for the preparation of GO for petroleum industry. The oil palm shells which are in large quantities has been made into activated carbon. Based on the Indonesian Palm Oil Association (IPOA) data in 2017, the Oil Palm Shell (OPS) is an agricultural waste currently measured at approximately 9 million tons per year. The OPS waste is widely used for charcoal briquettes, biofertilizers, and boiler fuel applications (Supriyanto et al., 2018). Hence, it appears to be the most promising and potential material to improve the technology and economic value of these waste.

2. METHODS

Preparation of the Graphite Biomass

Oil Palm Shell used as raw material to produced graphite biomass are obtained from one of the Palm Oil industries in Riau Province. The type of oil-palm shell chosen is the *Dura* type which has a shells thickness of 2-6 mm to obtain uniform properties. OPS was washed with water to remove the water-soluble contaminant and sun-dried for three days. After the drying process, the dried OPS was heated at 100 ℃ of temperature for 12 hours. Later, for the next 12 hours, it was pyrolyzed slowly at a temperature of 300 ℃. The pyrolysis sample was then sieved with a particle size of 300 mesh and activated by HNO3 for 24 hours. Then, it was filtered and cleaned using distilled water. Later, it was dried by the oven at a temperature of 100 ℃ to produce Graphite biomass.

Preparation of the Graphene Oxide Biomass

GO biomass was exfoliated from Graphite biomass using Ultrasound-assisted Liquid Phase Exfoliation (LPE) Methods. LPE work by exploiting ultrasound to extract individual layers (Monajjemi, 2017). The first process was the sonication that was the exfoliation process of 1.5 g graphite placed within the mixtures of 88.25 ml deionized water and 416.75 ml *acetone* for 4 hours (Yi et al., 2013). After sonication, the sample was centrifugated at a speed of 1000 RPM for 30 minutes at room temperature. Finally, the GO biomass powder was obtained by drying the sample at 100℃ of temperature for 3 hours.

The characterization of Graphite and GO biomass were done using three characterization techniques. First, to indicate the functional group of the Graphite and GO biomass, the Fourier Transform Infrared (FTIR) was used. Second, the UV-Vis Spectroscopy was used to conduct the optical properties analysis. Lastly, Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDX) was used at 15 kV of voltage. This characterization was used to analyses surface morphology and element of the Graphite and GO biomass.

Preparation and Characterization of the Drilling Fluid

The water-based mud (WBM) as the basic water-based mud fluid was prepared by mixing 22.5 g bentonite with 350 ml deionized water. This mixing process utilized Fann Multimixer which lasted for 20 minutes until homogeneous is achieved. The drilling fluid was age overnight for 16 hours at room temperature and lowered into a large (approximately 4 L) covered carboy. A stirring process was continued for 5 minutes afterward. For the proper comparison and analysis, WBM samples were prepared, which include WBM + 0.5 g Graphite and WBM + 0.5 g GO Biomass. Table 1 shows the composition of drilling fluid samples. The filtration properties, such as fluid loss and mud cake thickness, were measured using Low Pressure Low Temperature (LPLT) filter press test (Zamani et al., 2019). The fluid loss tests followed API guideline for water-based fluid. Tests were conducted in a standard API apparatus using Whatman quantitative paper filter. Filtration loss tests were conducted at 25 °C of temperature for 30 minutes with 100 Psi filter press supplied (API, 2003).

Table 1. Sample Component Mixture Amount

Substance	Samples		
	1	**2**	**3**
Deionized Water (ml)	350	350	350
Bentonite (g)	22.6	22.6	22.6
Graphite Biomass (g)	NIL	0.5	NIL
GO Biomass (g)	NIL	NIL	0.5

3. RESULTS AND DISCUSSION

Result

The characterization of Graphite and GO biomass was investegated. Figure 1 shows the FTIR spectra of Graphite and GO biomass. Graphite biomass has several essential peaks detected by FTIR patterns, such as -OH vibration at 3609 cm^{-1}, C-H stretching at 2804 cm^{-1} and at 771 cm^{-1}, C=O stretching at 1735 cm^{-1}, and C=C bond at 1591 cm^{-1}. The GO biomass spectrum shows an aromatic ring C=C that shifts slightly at 1596 cm^{-1}. Of particular interest is the appearance of the characteristic peak of carboxylate at 1722 cm^{-1}. In the spectrum also formed O-H bonds at 2620 cm^{-1} and 3631 cm^{-1}. C-O bonds at 1088 cm^{-1} and 1250 cm^{-1} indicate that the oxygen group in GO biomass.

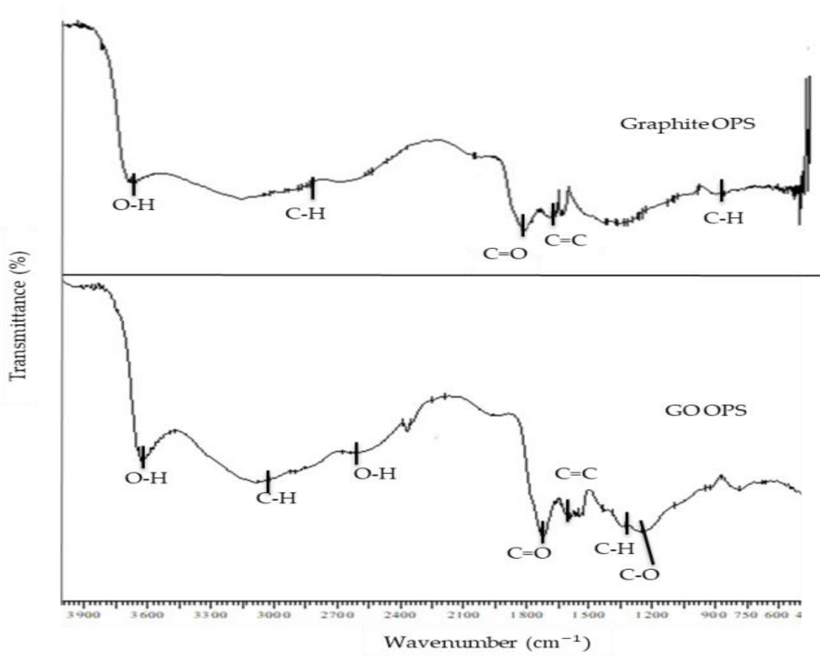


Figure 1. The IR Transmittance Spectrum of OPS Graphite and OPS GO

UV-Vis Spectrophotometer is mainly used to measure the transmittance or absorbance of the sample as a function of wavelength. Figure 2 demonstrates the UV-Vis graphs of Graphite biomass shown the weak absorption peak at 240 nm. Meanwhile GO biomass has an absorption peak at 269 nm.

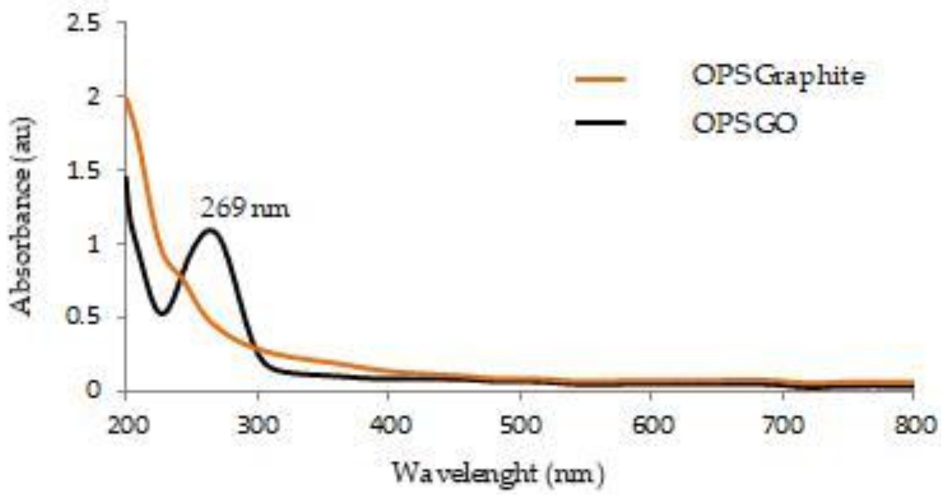
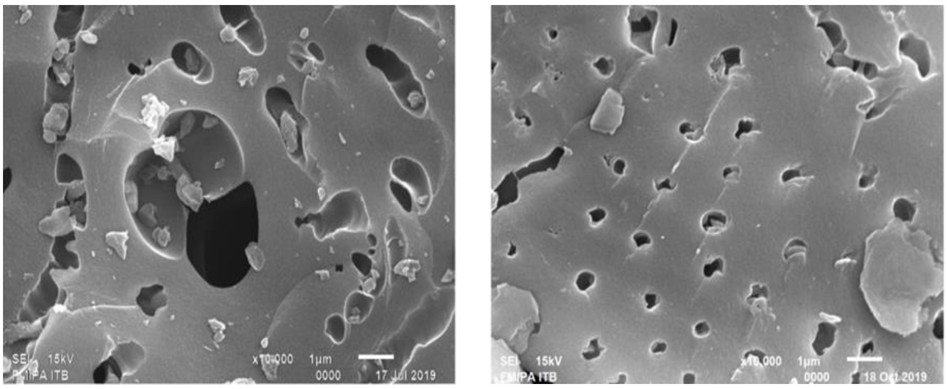


Figure 2. UV-Vis Graphs of Graphite and GO Biomass

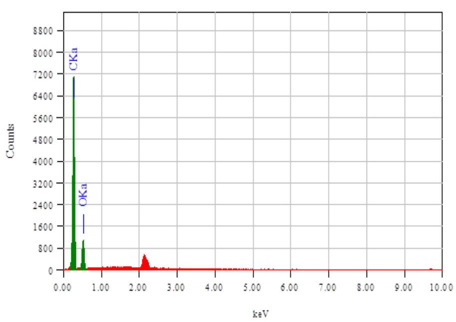
Scanning Electron Microscopy (SEM) was used to investigate the surface morphology of OPS Graphite and OPS GO. Figure 3 shows the surface morphology of Graphite and GO biomass with a magnification of 10000x. The Graphite has different sizes of pores with no pattern and unstructured distribution. Meanwhile, the pores formed in the surface of GO biomass surface have a similar size with a pattern, close proximity to each other, and oriented arrangement (Deng et al., 2017).



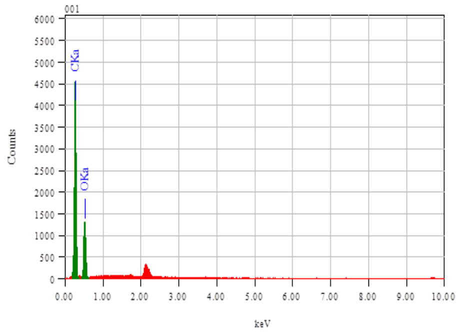
(a) (b)

Figure 3. SEM Image of (a) Graphite Biomass (b) GO Biomass

EDX characterization was used to analyze chemical characterization of Graphite and GO biomass. Figure 4 shows the results of the characterization. Based on Figure 4, Graphite and GO biomass consist of carbon and oxygen atoms. Table 2 shows the percentage of carbon- and oxygen-containing Graphite and GO biomass. The percentage of oxygen in GO (33.93%) is higher than the percentage of oxygen in Graphite (25.41%).



(a)



(b)

Figure 4. EDX Characterization of Graphite and GO Biomass

Tabel 2. The EDX data of Graphite and GO Biomass

Element	Carbon (%)	Oxygen (%)
Graphite biomass, 300 mesh	74.59	25.41
GO biomass	66.07	33.93

The effectiveness of the addition of Graphite and GO biomass on the decreasing filtration loss and mud cake thickness can be shown on Table 3. The addition of graphite dan GO biomass to the WBM has been successful in reducing fluid losses. However, the addition of GO biomass was more effective than graphite. The thickness of mud cake was also reduced by the addition of graphite and GO biomass.

Table 3. Filtration Loss and Mud Cake Thickness

Drilling Fluid	Filtration Loss (ml)	Mud Cake Thickness (mm)
Water Based Mud (WBM)	13.9	2
WBM + 0.5 g Graphite	12.3	1
WBM + 0.5 g GO	10.8	1.2

Discussion

Analyzing of the Graphite and GO Biomass

The FTIR spectrum of Graphite shows absorption peaks for the -OH bond at 3609 cm^{-1}, an aromatic ring C=C at 1591 cm^{-1}, C=O stretching at 1735 cm^{-1}, and C-H at 771 cm^{-1}. With these functional groups, the hybridization of the OPS biomass graphite allotrope is determined to be sp2. The infrared spectrum of Oil Palm Shell corresponds to the spectrum of coconut shell graphite reported by supriyanto (Supriyanto et al., 2018). Therefore, in this study, OPS graphite can be used as a source material for production of Graphene Oxide.

Furthermore, the GO biomass spectrum shows a slightly shifted aromatic ring C=C at 1596 cm^{-1} Furthermore, the GO biomass spectrum shows a slightly shifted aromatic ring C=C at 1596 cm^{-1}. Oxygen functional groups in GO biomass appear with O-H bonds at 2620 cm^{-1} and 3631 cm^{-1}. The hydroxyl group formation is consistent with the presence of oxides at the ends of graphene chains due to water molecules. The spectrum at 1735 cm^{-1} indicates C=O bond vibration caused by sp2 hybridization. Peaks at 1088 cm^{-1} and 1250 cm^{-1} represent C-O bonds. The FTIR characterization results of GO biomass from oil palm shell match the spectrum of GO from commercial graphite (He et al., 2015). Thus, GO biomass from OPS was successfully exfoliated from oil palm shell graphite using Ultrasound-assisted Liquid Phase Exfoliation (LPE) methods. The exfoliated GO biomass is water-soluble, producing a brown-colored solution, indicating its hydrophilic nature, a useful property for filtrate loss applications.

The UV-Vis spectrum of the graphite solution shows a weak absorption peak at a wavelength of 240 nm, consistent with the study by Sjahriza et al. (Sjahriza & Herlambang, 2021) on the UV-Vis spectrum of coconut shell graphite. After exfoliation from graphite to GO, the optical absorption peak of GO biomass sharpens at 269 nm with a broad edge reaching 300 nm. This indicates an energy transition from the ground state to the excited state of chromophores in the GO biomass solution. The absorbance peak at 269 nm is corresponding identifier of $\pi \rightarrow \pi^*$ transition in the double bond C=C of the OPS GO compound (Kigozi et al., 2020), absorbing energy in the ultraviolet wavelength range. The observed absorbance peak of GO biomass OPS falls within the range of GO material. The absorbance peak observed at wavelengths between 225 – 275 nm (4.5-5.5 eV) is characteristic of graphene or graphene Oxide (Eda & Chhowalla, 2010).

Based on the elemental analysis from EDX data, Graphite and GO biomass contain Oxygen and a high carbon. This indicates that the slow pyrolysis method used for graphite production was successful without other impurities. Other research results found graphite production with impurities such as Al, S, and K below 1% (Wachid et al., 2014).

Analyzing of the Effects GO Biomass in Control Filtration Loss

Based on the study results, the addition of Graphite biomass from Oil Palm Shell into Water-Based Mud (WBM) can reduce filtration loss compared to without the addition of Graphite biomass. The reduction is attributed to the presence of pores in Graphite biomass, so with the presence of graphite in the fluid, it binds the fluid within these pores. Fluid bound in the pores is less likely to be lost into the formation. This is consistent with previous research on filtrate loss with activated carbon, which has a molecular size of 40 micrometers (Mursyidah et al., 2019).

However, the reduction in fluid loss is found to be more effective when using well-exfoliated Graphene Oxide (GO) biomass derived from graphite biomass as an additive. The WBM with 0.5% GO biomass has successfully reduced filtration loss to 10.8 ml, approximately a 23% reduction compared to the filtration loss in the WBM sample. The prevention of fluid loss by GO biomass is based on kosynkin's opinion that during drilling there is high pressure (Kosynkin et al., 2012). The GO sheets in the fluid form a film on the formation sides to prevent filtrate from entering the formation. After reducing the pressure in the wellbore during recovery, oil and gas in the formation push the GO biomass sheets out of the wellbore walls (Kosynkin et al., 2012). The pressure applied in this study was 100 Psi (LPLT), allowing the GO biomass film to act as a barrier to prevent filtrate from entering the formation.

GO biomass can reduce fluid loss because it is hydrophilic, that is likes water. Therefore, the addition of GO biomass results in attractive forces between water molecules and GO biomass, forming a strong adhesive force. The formation of good adhesion to GO biomass will reduce the amount of initial fluid loss in the drilling process when the pressure is increased. A recent molecular dynamics study demonstrated the high affinity of GO to itself and other particles due to interparticle hydrogen bonding. The hydrogen bonding between the oxygen-containing functional groups on the surface of GO is likely responsible for the improved filtration characteristics. GO enters the micro pores in the formation and forms a very thin impermeable film layer, preventing fluid loss.

Oxygen reacts readily with bentonite in drilling fluid. This reaction makes the surface of the filter cake covered with the OPS GO biomass coating and the mud cake thickness is reduced from 2 mm to 1.2 mm. This process makes the formed filter cake more compact and prevents the fluid from entering the formation. More importantly, the nanometer thickness of the OPS GO biomass could also result in much thinner filter cakes than those obtained using graphite materials. The thickness of the filter cake of wellbore is directly and strongly correlated to the differential torque needed to rotate the pipe during drilling, to the drilling time and to drilling cost (Lyons, 1996). Therefore, adding OPS GO biomass to the drilling fluid can improve the filtration loss property and the quality of the filter cake. A thin layer of filter cake has a positive effect on drilling operations because it will reduce the power required to rotate the drilling bit thereby reducing usage energy and does not reduce the annular space between the drilling bit and the well formation.

4. CONCLUSION

The slow pyrolysis technique has successfully produced pure OPS graphite containing only C and O elements. Ultrasound-assisted Liquid Phase Exfoliation (LPE) methods have effectively exfoliated graphite layers into OPS Graphene Oxide (GO) biomass. Thus, OPS biomass has the potential to become a high-tech material. The application of OPS GO biomass as a lost control filtrate has been successfully discovered, resulting in a 23% reduction in filtrate loss and a decrease in mud cake thickness from 2 mm to 1.2 mm. From this research, Graphene Oxide Biomass shows great potential as an alternative material in preventing drilling fluid loss.

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