Rheological Study of Oil Palm Trunk Waste as Viscosifier Agent on Water-Based Mud

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***Abstract*— The Oil Palm Trunk (OPT) is one of the biggest agro-waste in Malaysia. OPT, is rich with cellulose which has potential of becoming viscosifier agent in designing Water-Based Mud (WBM) by improving the rheological properties of WBM. In this study, the cellulose was extracted by using two methods which are Method 1 (Chlorination-Bleaching Process and Mercerization Method) and Method 2 (Dewaxed-Alkaline-Delignification method). The characterization of OPT cellulose is defined by FTIR analysis while the content of cellulose product is defined by using Weighting Method. In this experiment, the mud samples were formulated by presence of different weight of cellulose applied. The production method and determination of rheological properties (Viscosity, Gel Strength, Yield Point and Filtration Test) were carried out based on API mud production standards. From the analysis of the experiment results, Method 2 yields more percentage of cellulose by 43.05% while the Method 1 only yield percentage of cellulose by 37.63%. Based on FT-IR Spectrometry results, it showed that Method 2 mimicked the behavior of HEC cellulose (commercial product) which is widely used as viscosifier agent for the WBM. In addition, both Method 2 and HEC cellulose have similar functional group that appear at same spectral peaks from the FT-IR analysis. In conclusion, Method 2 cellulose shows the best and almost following with the API standard for WBM on rheological properties.**

***Keywords*— Cellulose, Oil palm trunk (OPT), Rheological properties, Water-based mud (WBM), Fourier Transform-Infrared (FT-IR).**

I. INTRODUCTION

According to American Petroleum Institute, drilling fluid is defined as circulation fluids used in drilling operation. There are 3 major types of drilling fluids such as Water-Based Mud (WBM), Oil-Based Mud and Synthetic-Based Mud. The major function of drilling fluids is to clean the wellbore from drilling cuttings [1], acts as cooling and lubricant the drill string and drill bit from damage. The WBM has the advantages of shear thinning, high true yield strength, good bit hydraulics, reduced circulating pressure losses, improve borehole stability and economical and eco-friendly [2]. The capability of WBM is determined based on the rheological behavior of the WBM such as, mud weight, pH, plastic viscosity, yield point, and gel strength.

Rheological properties are described by rheologic model. In Bingham Plastic Model, the term plastic viscosity (PV) is used as an indicator of the size, shape, distribution and quantity of solids; and the viscosity of the liquid phase; and yield point (YP) is a measure of electrical attractive force in the drilling fluid under flowing conditions. PV and YP are two very important drilling fluid parameters, and both can be calculated from mud viscometer data. The additives are needed in designing WBM to formulate and determined the rheological properties so the WBM can used at different reservoir condition.

The additives include viscosifiers, viscosity reducers, weighting materials, and pH control additives. Currently, drilling operation mostly used common additives polymers into the WBM fluid for the such as Carboxy-Methyl Cellulose; CMC, and Hydroxy-Ethyl Cellulose; HEC [3]. Oil palm trunk (OPT) become one of contributor to agricultural waste material from palm oil industry in Malaysia [4].

It is known as non-wood lignocellulosic material consists two major components which are Parenchyma and Vascular bundles as tissues. The vascular bundles provide support to cells wall and transport water and the nutrients and it is consisting of xylem, fibers, sieve tubes, vessels, axial parenchyma, protoxylem, stegmata and companion cells. The study also proved that the OPT has richest contain carbohydrates that form of sugar-containing cellulose, starch, hemicellulose and lignin [5].

The chemical composition of cellulose in OPT consists around the range of 29% to 50% and the lignin was 20% to 25% and proven that the cellulose provided is a strongest mechanical strength to plant tissue by the unbranched-beta (1-4) linked D-glucopyranose units of polymer chains built-up in cellulose structure.

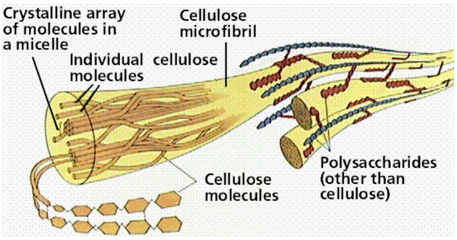


Figure 1: Cellulose Molecules in Fibers of OPT [3].

Cellulose is renewable polymer resource and the cellulose apparent is caused by existing lignocellulosic material by being part of woods trunk components [6]. Cellulose has a high capability in water absorption capacity. The higher concentration of cellulose increases the viscosity of the drilling fluids. In this study, the objectives use of cellulose as viscosifier agents are to evaluate the effects of different weight of cellulose to the rheological properties, the effects of temperature on rheological properties of WBM by using OPT cellulose as viscosifier additives and the comparison in term of rheological behavior between OPT cellulose and commercial viscosifier agent (HEC-polymers).

II. METHODOLOGY

A. Materials

In this study, the OPT waste was obtained from the Forestry Research Institute of Malaysia (FRIM). The sample then dried in the oven for two days under the temperature of 70°C. This OPT fibers then ground and sieved for the range of 710-150 µm sizes.

The samples later were stored in airtight polybag at room temperature. The chemical used were Sodium Chlorite ($NaClO_2$), Dilute Acetic Acid, and Toluene, Methanol, 95%-Ethanol, 10% and 20% NaOH.

B. *Methods*

- Method 1 (Chlorination-Bleaching Process and Mercerization Method)

There are two parts separate process which are Chlorination-Bleaching process and Mercerization method. First part, the OPT was ground using grinder about sized 500µm then sample measured about 20g then rinsed with tap water to remove the dust. After added 4mL (5%) acetic acid and 8g Sodium Chlorite added into beaker then left overnight. The sample again been then rinsed up with tap water to remove thee yellow yellowish residue and become odorless. For the second part, the present of holo-cellulose from part 1 soaked with 80mL (17.5%) Sodium Hydroxide and stirred with gas rod. In addition, about 40mL of NaOH added 3 times within interval 5 minutes until NaOH treatment total time 45 minutes. Later, 240mL distillation water added then 800mL (8.3%) NaOH into cellulose sample for 5 minutes and rinsed with tap water again. The alkaline cellulose was cleaned up with (10%) of acetic acid for 5 minutes. The cellulose sample was filtered, washed and rinsed then let dried overnight in oven at 80°C lastly secured in the air-tight polybag.

- Method 2 (Dewaxed-Alkaline-Delignification Method)

Method 2 can extract the cellulose from the OPT waste by the delignification process (removal lignin treatment). Firstly, 5.8g grinded OPT cellulose filled up to cellulose thimble for Soxhlet apparatus with toluene-ethanol (2:1, v/v). The process took about 6 hours long at 100°C, and later the obtained powder was dried 2 days at room temperature. The delignification treatment conducted by applied acidified Sodium Chlorite ($NaClO_2$) at 75°C for 1 hour. The product was then treated with 2wt% KOH, 5wt% KOH at 90°C then filtered and rinse with water until pH shows natural and the chemical purified cellulose (CPC) stored and lastly the sample was sealed in the air-tight polybag.

- WBM Procedure

First step in designing the WBM was by preparing the 10g bentonite solution, in 350mL of tap water as based fluid. Later, the solution was left overnight. All additives are added into mixer within interval of 5 minutes each. The additives that were added in the WBM formulation are Sodium Chloride (NaCl), Xanthan Gum, Sodium Hydroxide (NaOH), Barite and the different weight of OPT cellulose.

C. *Characterization Methods*

- Fourier Transform Infra-Red (FT-IR) Spectroscopy

The IR spectra were obtained from a Perkin-Elmer FT-IR model Spectrum 100 Series Universal Attenuated Total Reflectance (UATR) techniques. The absorption bands were measured in (cm-1) within the wavelength region from 400 to 4000 cm-1. The sample powder simply put into the sample detector. The expected result is the wavenumber of functional group such as OH-alcohol group detect which is the cellulose components have high OH group bonding.

III. RESULTS AND DISCUSSION

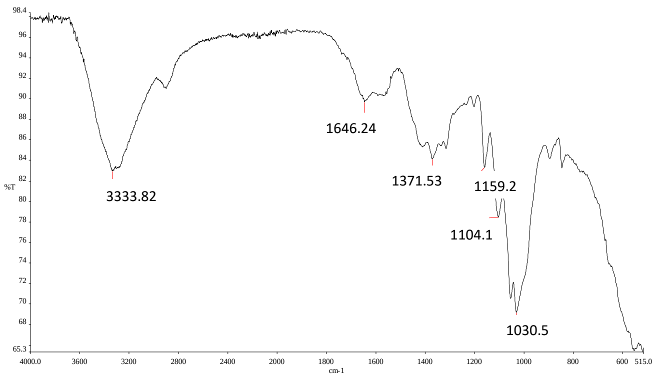
A. *The Yield Percentage of Extraction Cellulose from OPT Wastes*

The delignification process is important in removal of lignin from OPT waste. In Method 1 and Method 2 are applying this process to extract the cellulose from OPT waste. Method 2 yield more cellulose percentage from OPT waste; 43.05% (lignin and other component 56.95%) as there is a heating process applied in this method. While Method 1 yield cellulose with only 37.6% (lignin and others component are 62.40%) and does not involved any heating process. The heating process makes the extraction more efficient in separating the cellulose from the other component in OPT waste sample (lignin). By using both methods, the percentage of lignin and other component in OPT waste is about 56.95% and 62.40% for Method 1 and Method 2, respectively. In which, it supported by [7], that the percentage range of delignification process will removed 56-62%. The yield cellulose obtained by both methods is summarized in Table 1.

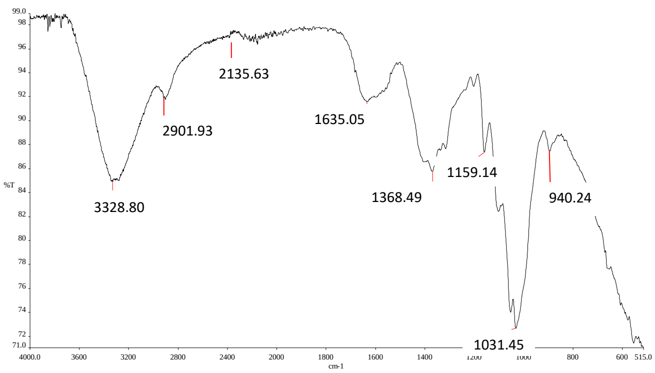
Table 1: Yield Percentage of Extractive Cellulose

Extraction Method	Method 1	Method 2
Weight of OPT (g)	40.0	40.0
Final Weight of Cellulose (g)	15.05	17.22
Percentage Yield Cellulose (%)	37.6	43.05

B. *The FT-IR Spectrometry Results*



(a)



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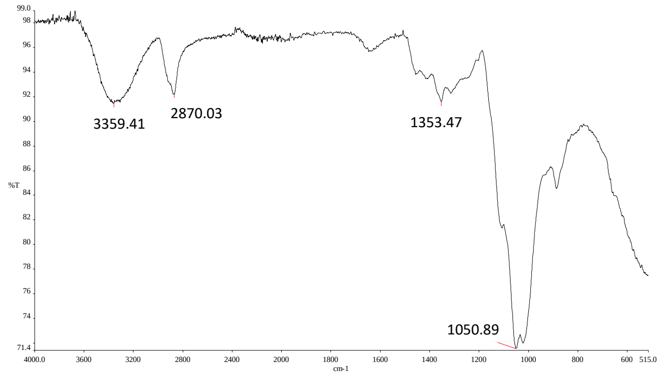

(c)

Figure 2: FT-IR Analysis for (a) Method 1, (b) Method 2, (c) HEC Commercial Cellulose.

Figure 2 (a), (b) and (c) shows the results analysis of OPT and HEC cellulose by using FT-IR spectrometry. One of characterization of cellulose is based on present of OH functional group, and the absorption band peak of OH-group is observed near 3400-3200 cm^{-1} [8]. All Method 1, Method 2 and HEC cellulose shows the same spectral band peak at the range of 3400-3200 cm^{-1} but different in term of transmitted intensity for 82% (Method 1), 85% (Method 2) and 91% (HEC-commercial).

The absorption band peak at range of 3000-2800 cm^{-1} shows the CH-stretching vibration which is corresponded to CH stretching vibrations and corresponded to the aliphatic of two parts in polysaccharide which are cellulose and survived hemicelluloses. Moreover, the comparison between methods, Method 2 and HEC commercial shows the same absorption band peak but Method 1 no observed spectral band peak at that range. There is possibility of CH-stretching did not happen in Method 1 compare to Method 2 and HEC commercial. The absorption spectral band peak at region 1400-1100 cm^{-1} shows in Figure 2 (a), (b) and (c) are the CH deformation in cellulose and hemicellulose, C-O-C vibration in cellulose and hemicelluloses and CO vibration in cellulose and hemicelluloses occurred.

However, the CH deformation in cellulose and hemicellulose was not occur in HEC commercial cellulose due to its specialty of HEC as the derivative cellulose which is stable formation in the HEC. The results from Method 1 and Method 2 shows the OPT cellulose are less efficient compared to HEC cellulose because they are easily form a CH deformation in cellulose and holo-cellulose which prove that the HEC commercial can give a better performance compare to OPT cellulose waste. Overall, all Method 1, Method 2 and HEC commercial show same trend of spectral absorption band peak near 1000-900 cm^{-1} which is the CO vibration in cellulose and hemicellulose. In conclusion, the comparison between Method 1, Method 2 and HEC commercial shows the spectral band of Method 2 are mimicking the spectral band of HEC commercial. Thus, the properties of cellulose from Method 2 slightly same as the HEC commercial.

C. Water-Based Mud Experimental Results

1) Rheological Analysis

The rheological analysis was studied to observe the behaviour of WBM in term of its rheology properties (plastic viscosity, yield point, gel strength, mud weight and pH) and the effects of different weight of cellulose and temperature used toward rheological behaviour of WBM.

- Mud Weight and pH Results

The rheological test for mud weight and pH have conducted for mud sample 1, 2 and 3 (OPT-Method 1, OPT-Method 2 and Commercial cellulose). 3 different weight of OPT cellulose and commercial cellulose (4g,6g and 8g) were used in this rheological test. The mud weight for this mud is constant at 9.70ppg based on the calculation in designing WBM, as referred to API drilling mud production standards [9]. Table 2 below shows the results of mud weight and pH test of the formulated WBM.

Table 2: Mud Weight and pH of WBM.

Parameter/Weight	Method	4g	6g	8g
Mud Weight (ppg)	Method 1	9.7	9.7	9.7
	Method 2	9.7	9.7	9.7
	HEC	9.7	9.7	9.7
pH	Method 1	10.74	10.48	11.55
	Method 2	10.75	10.8	11.56
	HEC	10.8	10.9	11.6

The pH results showed increasing trend in values of pH as the weight of OPT cellulose and HEC commercial cellulose were increased. The mud become more alkaline because the present of OH functional group as shown in FT-IR result and both methods shows almost the same value as HEC commercial cellulose.

2) Different Weight of OPT Effects on Plastic Viscosity, Yield Point and Gel Strength at Reservoir Temperature 48.8°C

- Plastic Viscosity (PV)

The plastic viscosity is a slope of shear stress against shear rate, representing as viscosity of mud. It can be calculated from dial reading of 600 rpm and 300 rpm. Figure 3 below shows the effects different weight OPT and HEC cellulose toward the plastic viscosity behavior.

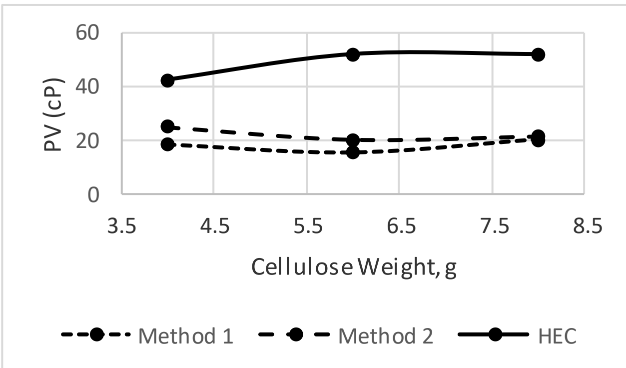

Figure 3: Effects of Different Weight OPT Cellulose and on PV Behavior.

Method 1 OPT cellulose gives a slightly decrease from weight of 4g to 6g and starts to increase from 6g to 8g of OPT cellulose. While Method 2 also shows same trends of graph as Method 1 which is decreases but increasing slowly. The HEC commercial cellulose shows different trends of graph from OPT cellulose by slowly increases then constant value of PV. The optimum range of plastic viscosity is between 16cP to 22cP [9]. Thus, the PV from Method 2 is higher than Method 1 with 20cP value of PV due to present of intermolecular interaction due to hydrophobic alkyl substituents effects the flow behavior in term of viscosity properties by absorb OH functional group in alkyl substituents easily combined and reacts with fluids [10].

- Yield Point (YP)

Based on Figure 4 below, Method 1 and Method 2 show the increment trend of YP from 47.4 to 85.7 (lb/100ft^2) and from 45 to 59 (lb/100ft^2) repesctively as the weight of OPT increased from 4g to 6g. However, the YP performance start to decreased when the weight OPT cellulose increased from 6g to 8g. from Figure 4, the pin-point of the optimum YP was observed when the performance of YP started to degrade which is at 6g of OPT cellulose. The HEC commercial gives a highest value of YP compare to Method 1 and Method 2. Theoretically, the increament of YP values effects by the increase of viscosifier's weight or concentration (as supported by [9]) the viscosifier agent such as HEC will re-arrange and react with presence of water and other additives in WBM. Hence, the viscosity will increase in parallel with YP values. However, for both Method 1 and 2, at 8g additional of cellulose, the YP decreased. This may due to the errors happened during preparation of the WBM at 8g of OPT waste cellulose. The summarized data of yield point as shown from Figure 4.

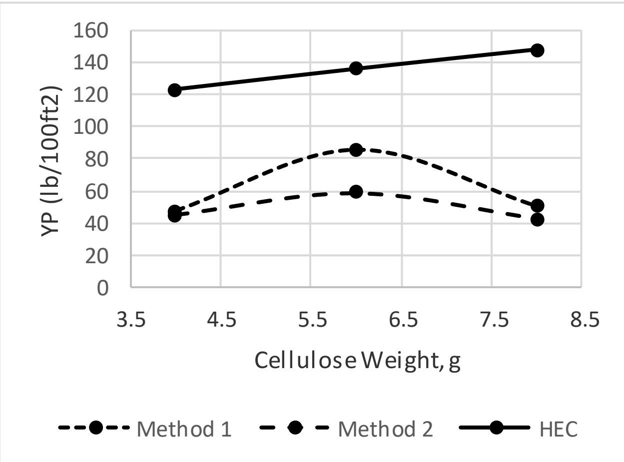

Figure 4: Effects of Different Weight OPT Cellulose and HEC on YP Behavior.

- Gel Strength

The gel strength refers to the shear stress required to initiate flow after static periods of time. In this study, the gel strength taken at the time of 10 minutes. The results from Figure 5 shows that increase weight of Method 1, Method 2 and HEC commercial cellulose will also increase the gel strength values. While the HEC commercial cellulose shows the highest values and its value increases in term of gel strength for 4g, 6g, and 8g by 23, 26, and 27 (lb/100ft^2). The Figure 5 below, shows the performance of gel strength for both Methods increase trends when the value of OPT Cellulose and HEC weight increase. In the Figure 5 shows results for Method 2 is increases values of gel strength parallelly with increases the weight of OPT cellulose.

Theoretically, the increasing in value of gel strength will ease the drilling fluid bring cutting into surface, but in most follow certain range value because the over range value of gel strength also give effect to the hard to drilling cutting pumping out to the surface from wellbore [3].

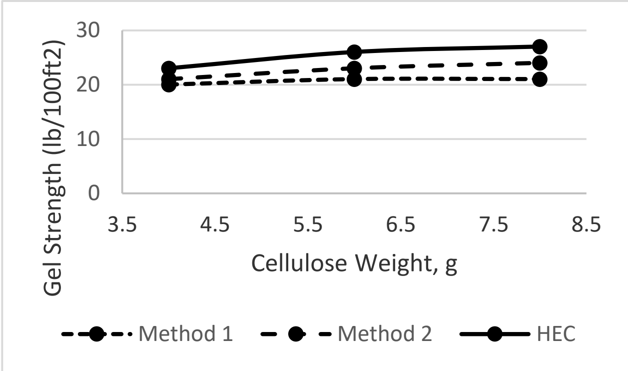

Figure 5: Effects of Different Weight OPT Cellulose and HEC on Gel Strength (10 minutes) Behavior of WBM.

3) Different Temperature Condition Effects on 4g of OPT Cellulose and HEC Commercial Cellulose in term Rheological Behaviors.

- Plastic Viscosity (PV)

The plastic viscosity behavior was observed based on the effects of different temperature in term of rheological properties of WBM performance. Generally, the temperature selection based on the real reservoir condition which is 48.8°C for normal temperature and 60°C for high temperature by supported by [2].

Based on Figure 6, it shows the result of PV affected by increases in temperature. The trend of graph in Figure 6 of Method 2 and HEC cellulose are same; which decrease in PV value while the temperature increased. For Method 1, the PV value increase as the temperature increase from 48.8°C to 60°C. Meanwhile, at temperature of 70°C. show that the PV performance decreased to 23.3 cP. There is possibility, by introducing heat (increasing temperature) the delignification process occurred, in which, OPT waste will yield more cellulose content afterward. Thus, the yield percentage of cellulose increase, which interact with WBM. Even cellulose and HEC will not easily degrade at high temperature, but another component in the mud will degrade due to increase of temperature.

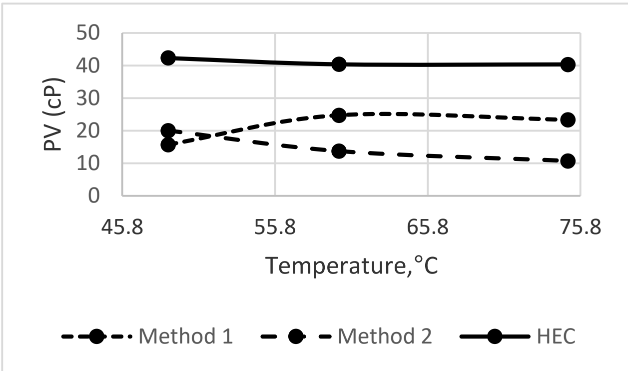

Figure 6: Effects of Temperature to PV of WBM Rheological Behavior.

- Yield Point (YP)

The rheological behavior (YP) is affected by the different temperature applied to the WBM. Figure 7 below shows about the effect of YP behavior while increasing the temperatures. The OPT cellulose from direct method shows the decrease in YP behaviour due to increasing the temperature. The OPT cellulose from Method 1 shows the decrease in YP behavior due to increasing the temperature from 85.7 lb/100ft^2 at temperature of 48.8°C, to 85.7 lb/100ft^2 at 60°C and 76 lb/100ft^2 at 75°C. It is similarly in term of YP performance which is decreasing while increasing in temperatures. At temperature of 48.8°C, the YP value is 59 lb/100ft^2 decreased slowly to 54.7 lb/100ft^2 at temperature of 60°C and continuously decreased to 52.3 lb/100ft^2 at temperature of 75°C. Meanwhile, the HEC cellulose give a different trend of graph from Method 1 and Method 2. During the temperature of 48.8°C, the YP value is 123.3 lb/100ft^2. Then, it was decreasing rapidly to 85.67°C as it reaches temperature of 60°C; but suddenly increase high at temperature of 75°C by 124.7 lb/100ft^2. There is possibility that error may occurred during formulating the WBM or possibility of degradation of HEC occurred during that time.

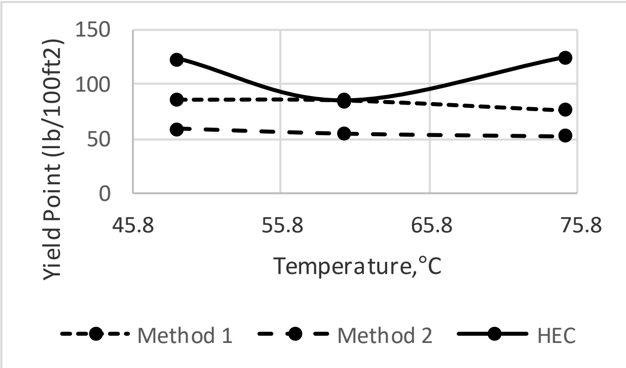

Figure 7: Effects of Temperature to YP of WBM Rheological Behavior.

- Gel Strength

In this experiment, the gel strength behavior is experimented by applied varies temperature replicated to the reservoir condition (48.8°C, 60°C and 75°C). Based on Figure 8 below, the behavior of gel strength for Method 1 and Method 2 give slightly a same trend while the HEC cellulose give different gel strength. This may due to the ability of HEC degraded until it exceeds limit (100°C). Method 1 and Method 2 cellulose can still counter with the increasing of temperature, but it starting to decrease the gel strength behaviour of mud at temperature of 60°C due to the integrated mud and cellulose starts to degrade at temperature of 60°C. The behavior of Method 2 and HEC are almost the same based on Figure 8. Thus, the Method 2 OPT is sufficient as viscosifier agent because OPT cellulose of Method 2 has almost same gel strength behavior.

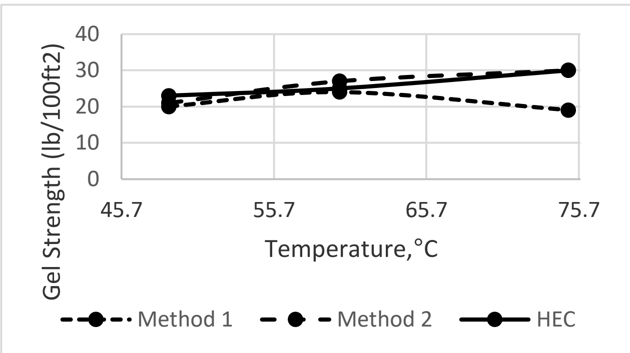

Figure 8: Effects of Different Weight OPT Cellulose and HEC on Gel Strength (10 minutes) Behavior of WBM.

4) The Effects of Varies Weight OPT and HEC Cellulose on WBM Behavior by Filtration Test.

- Filtration Test.

Measurement of filtration behavior and the filter cake are fundamental to the treatment and control of drilling fluid [9]. The Figure 9 below, shows the filtration fluid by comparing different method with varies weight of OPT and HEC cellulose used in this experiment. The results show the performance of mud filtration for Method 1 and Method 2 decreased when increasing in weight of OPT cellulose. Besides, Method 2 shows the fluid loss in WBM decrease from 12.6mL to 12.4mL due to increased weight of cellulose. The optimum for mud filtration for Method 2 at 6g weight of OPT cellulose. Meanwhile, HEC cellulose have same trend with Method 2 cellulose in term of fluid loss from WBM. The optimum mud filtration for HEC cellulose at 9.6mL of fluid loss at 4g weight of cellulose. The mud cake for all 4g, 6g and 8g weight of OPT and HEC cellulose shows the best performance which is by 1/32 inches (0.794 mm) in which is optimum for mud cake filtration according to API standard for drilling fluid design stated.

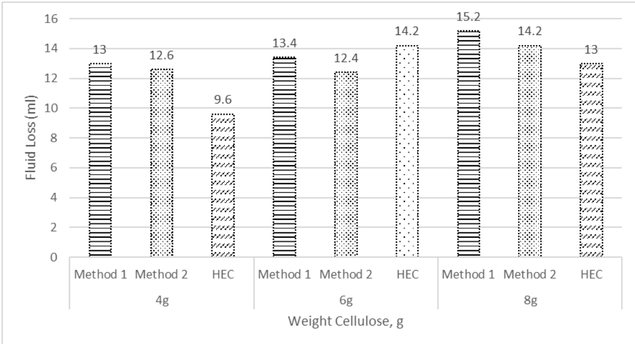

Figure 9: Effect on Filtrate Volume in Varies Different Weight of OPT and HEC Cellulose.

IV. Conclusion

In summary, the study has two parts which is separately conducted. First part is the extraction of cellulose from OPT waste by Methods 1:(Chlorination-Bleaching Process and Mercerization Method) and Method 2:(Dewaxed-Alkaline-Delignification Method). By comparing by these two methods, the Method 2 has ability and sufficiently yielding more percentage of extraction cellulose from OPT waste compared to Method 1. The characterization of cellulose has been done successfully by using FT-IR Spectrometry which is give a result in term of functional group that present on the cellulose itself and achieved the first objective in this study. Second part is the rheological properties been tested by determination the effect of varies different weight of OPT and HEC cellulose in rheological properties of WBM. In this study, there are two parts which is the effect of weight cellulose and the effect of temperature to the rheological properties for WBM. Based on the experimental parts of rheological properties for the effect of varies weight of cellulose on the PV, the Method 2 cellulose gives a good range of results as compared to the standard range of PV for the WBM by API Recommended Practice 13B-1. In addition, it shows the best performance at weight of 8g with 21.3cP. While for HEC cellulose as viscosifier agent, despite showing the highest PV value (by increasing weight of cellulose), the PV value also increase but it does not meet the range of PV standard for WBM. Based on YP behaviour test, Method 2 also shows the best yield point results at 6g weight of cellulose compared to Method 1 and HEC commercial.

Next, the gel strength test shows the increase gel strength values by increasing weight of OPT and HEC cellulose. Overall, OPT cellulose (Method 2) shows the best performance in this rheological test. Thus, the objective to analyze effects on rheological properties by applied different weight of OPT waste and HEC commercial are achieved. Lastly, the effects of temperature at 4g of OPT cellulose was conducted to study the rheological properties performance. Generally, the performance of all OPTs and HEC cellulose start to decrease in performance while increase the temperature. The increase of the temperature does affect the performance of the mud as the cellulose and components in WBM also start to degrade. The fluid loss at 4g of cellulose shows the lowest results which is good in term of drilling operation performance. Besides, the mud cakes for all 4g, 6g, and 8g of cellulose shows good result by 0.79mm less than 0.79375mm. As conclusion, the OPT cellulose from Method 2 was the best viscosifier agent to obtain the best rheological and filtration performance as compared to Method 1.

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