

Study of biomass composition and porosity of raw/torrefied Oil palm frond

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Abstract— In this recent decades, Malaysia face challenges in utilizing fossil fuel as a source of energy. This is due to declining supplies and adverse environmental effects. Therefore, an alternative option has been taken to solve the problem. The renewable energy plays the main role as sources of energy and replace fossil fuel utilization. Among the alternatives, biomass fuels have high tendency and potential in producing the renewable energy which is the fourth most prevalent energy source after coal, oil and natural gases. In this study, the potential of oil palm frond as a biochar feedstock for bio-energy purposes are being explored. Among the properties of OPF to be studied are biomass composition, the characteristics of raw/torrefied oil palm frond (OPF) and other physico-chemical properties. For example, proximate analysis, ultimate analysis and calorific value analysis. Study also is conducted to analyse the porosity of OPF. Proximate analysis was performed via thermogravimetric analysis whereas the biomass composition was determined via Technical Association of Pulp and Paper [TAPPI] method. For the determination of surface area and porosity of OPF, Brunauer Emmet Teller [BET] method is introduced as a method to measure the surface area of oil palm frond. As for elementary analysis, during increasing of torrefaction temperature, the elemental C contents gradually increased and the H and O elements gradually decreased. Then, higher carbon content normally leads to higher calorific value. Raw OPF contained higher volatile matter followed by ash, fixed carbon and moisture content. In raw OPF fibre composition, the highest percentage of lignocellulosic is cellulose (44.17 wt%), followed by hemicellulose (34.83 wt%), lignin (21.00 wt%), and extractives (20.26 wt%). For BET surface area, the surface area will change into different size at a different temperature for HT60 minutes but not in the case of HT30 minutes. As a conclusion, the OPF is observed to be a better feedstock of biomass when subjected to torrefaction, so that it can take place the utilizing of fossil fuel to produce energy and it can possibly replace fossil fuel as an alternative biofuel.

Keywords— oil palm frond residue (OPF), biomass composition, ultimate analysis, proximate analysis and porosity analysis.

I. INTRODUCTION

There is a certain limitation of oil palm frond as a source of energy in biomass. The raw biomass of OPF is classified as low quality of fuel due to structural heterogeneity, high moisture content, non-uniform physical properties, low energy density, hygroscopic nature, and low bulk density. The few weaknesses obtained need to be overcome before it can be converted into a useful energy source [1].

In order to handle these problems, the properties of raw biomass need to be improvised. The thermal heating process known as thermochemical pre-treatment process is carried out. Then, an

investigation of chemical elemental characteristics of biomass fuels might be beneficial as to identify suitable energy conversion technologies.

Prediction of high energy value of OPF can be done, whereby in this study, few methods are being done which are biomass compositional study and other physico-chemical biomass characterization. Therefore, the characterization of these oil palm fronds OPF in terms of proximate and ultimate analysis, energy content, surface area and adsorption capacity are carried out to assess its potential as source of energy fuel. The results shown include the characteristics of oil palm frond biomass in terms of carbon content, calorific value, surface area and adsorption capacity.

II. METHODOLOGY

A. Materials

The sample are oil palm frond (OPF) residue which was collected from oil palm plantation in Klang, Selangor. The sample used are in the form of pellet. For pore analysis, all the pellets were grinded first into particle size <212 micrometer. by using mortar grinder. The ground sample was then collected and stored in air-tight container.

The three main apparatus used in this study are:

- UV spectrometer
- Soxhlet apparatus and extraction set up
- Surface Area Analyzer (BET) 3FLEX Micromeritic
- Elemental analyzer CHNS Bomb Calorimeter (TGA)

All the equipment can be found at the laboratory of Faculty Chemical Engineering UiTM Shah Alam.

B. Procedure

1) Characteristics of raw/torrefied oil palm frond (OPF)

The characteristics of raw and torrefied OPF samples are analysed by using the proximate analysis and ultimate analyses. The thermogravimetric analyser (TGA), was selected as the equipment was used to perform the proximate analysis of oil palm frond. The torrefaction process was performed with different temperatures ranging from 200°C to 300°C. For every test, 10 mg of sample was measured and loaded into an alumina crucible. Then, it was mounted in the heating chamber of the thermogravimetry (TG). Basically, nitrogen gas was used as a carrier gas and transported into the TG so that the material was torrefied in an inert environment without oxidant [2].

2) Determination of extraction

Raw OPF sample was weighted at 21g. The dried sample were extracted with toluene/ethanol with the ratio of (2;1, v/v) for 6 h in a soxhlet apparatus. The fiber to solvent ratio was 1:10 g/mL⁻¹ where 21 g of OPF sample mixed into 210 mL of solvent. The solvent was a mixture of 140 mL of toluene and 70mL of ethanol. After that, the sample were dried in an oven for 24 hours at 60°C, then reweighted again. The residues were subjected to extraction with 95% ethanol for 4 hours in a Soxhlet apparatus and then with

water at 100 °C for 2h. Thus, the sample now was free from extractives, ethanol soluble and water soluble [3].

3) Determination of lignin content

After hydrolysis with 72% sulfuric acid as per required by TAPPI T222 om-98, klason lignin is the insoluble residues retrieved.

An amount of 0.1g raw OPF fibre was weighted and added to a mixture of 5mL of 0.02 mol/L KMnO₄ and 20 mL of 2.0 mol/L H₂SO₄, followed by stirring them well for 3 minutes. Then, separated the solid sample was separated from the solution through filtration. The filtrate was then and measured using UV-Spectrometer at 546 nm of wavelength. The One-Point calibration method was carried out to determine the value of Kappa Number K which is shown in Eq (1).

$$K = \frac{a (A_o - A_e)}{w \quad A_o} \quad \text{Eq(1)}$$

Where K is Kappa Number, a is the volume of KmNO₄ used in the solution, w is weight of moisture free sample used, A_o is spectral intensities at time t = 0 which is the time before sample is added and A_e is spectral intensities at the end of the reaction. Determination of lignin was done by using the Eq (2) [4].

$$\text{Lignin (wt\%)} = 0.15K \quad \text{Eq(2)}$$

4) Determination of hemicellulose and cellulose

Hollocellulose is a combination between cellulose and hemicellulose, WISE method was used to determine the percentage of cellulose and hemicellulose of OPF. For preparation of holocellulose, about 4.0 g of the ground OPF fibre was measured. The measured sample was mixed into 50 mL distilled water and treated with 2.0 mL acetic acid and 5 g sodium chloride.

Then, the mixture was soaked in water bath with temperature 70°C for 4h. After that, measured to 2g of the produced holocellulose was measured and treated again with 50mL of 17.5% sodium hydroxide solution and the 70 mL distilled water was added into the mixture. As a result, hemicellulose was separated from the holocellulose and leaving the only cellulose. The insoluble cellulose was filtered and washed with 50 mL of 8.3% sodium hydroxide. The filtrate then was dried at 80°C for 24 hours. The determination of cellulose is determined by Eq (3). [5]

$$\text{cellulose (wt\%)} = \frac{W_i - W_f}{W_i} \times 100\% \quad \text{Eq(3)}$$

Which W_i is initial weight of sample and W_f is final weight of sample. Then, weight percentage of hemicellulose was determined by Eq (4).

$$[\text{Hemicellulose} = 100\% - [\text{lignin (\%)} + \text{Cellulose (\%)}] \quad \text{Eq(4)}$$

5) Porosity analysis

For porosity analysis, 11 samples of raw and torrefied OPF was grinded into the small size, then the sample was sent to the laboratory. The experiment was run by the lab instructor and it will take almost two weeks to get the result obtained. The apparatus used is Surface Area Analyzer (BET) 3FLEX Micromeritic.

		(°C)	(HT/min)
1	RS-2	150	30
2	TOPFP-200°C	200	
3	TOPFP-225°C		
4	TOPFP-250°C		
5	TOPFP-275°C		
6	TOPFP-300°C		
7	TOPFP-200°C		
8	TOPFP-225°C		
9	TOPFP-250°C		
10	TOPFP-275°C		
11	TOPFP-300°C		



Firsly, to start up the power supply and computer were switched on. Hydrogen and Nitrogen cylinder valves were opened and located the ball valves after pressure regulators. The pressure regulators were set to 10 psi. Then, to degass the sample, weighed the blank sample tube and seal red. Sample was inserted into the blank tube by using funnel and again the sample tube with inserted sample was being weighted again. The weight was recorded before degassing. Put the sample tube at degasser and required data was filled to start the degassing process. Smartprep degass was selected at the unit 1 followed by file name and correct port. Then, start button was selected to degases the sample. Then, Sample was taken out from the sample tube after the degassing process. All data is recorded as the sample after weighted the degassed sample. During an analyzing of sample, sample tube was plugged in at the analysis port and the dewar was filled with liquid nitrogen. At unit 1, clicked the sample analysis and the file name was filled with previous fill name sample. Then, weight blank tube and after degassing were key in into the file name. The shiel/cover analysis port were hung and start and next button were clicked. Then, automatically analysis was performed. Lastly, to clean the sample. The sample cell was empty and cell was cleaned by sonification with DI water.

III. RESULTS AND DISCUSSION

A. Characteristics of raw/torrefied oil palm frond (OPF)

1) Ultimate analysis

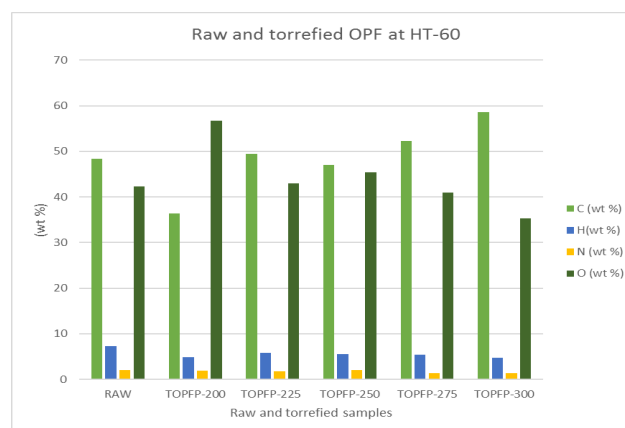


Fig. 1.0 (a) Ultimate analysis of raw and torrefied OPF at HT-60

Table 1.0 Sample of raw and torrefied OPF

No.	Name of samples	Condition	Heating time
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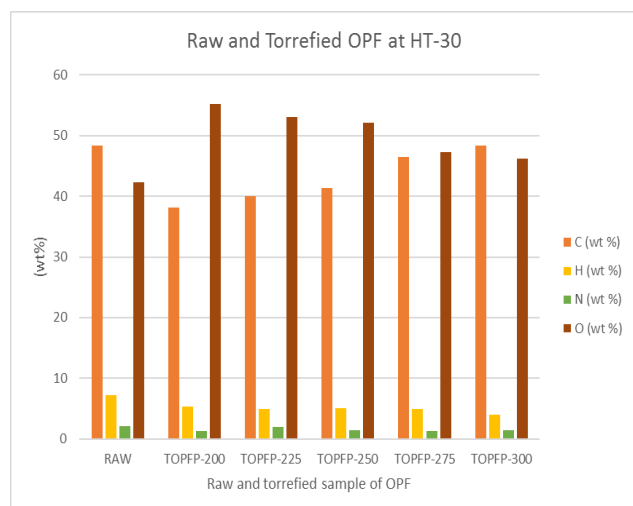


Fig. 1.0 (b) Ultimate analysis of raw and torrefied OPF at HT-30.

The ultimate analysis was done to evaluate the basic chemical components which determine the importance of any fuel. Based on Fig. 1.0 (a) and Fig. 1.0 (b), the carbon content at holding time 60 is increase but inconsistently whereas at holding time 30 minutes, the carbon content gradually increased. Notably, high carbon content is necessary for fuel application where it indicates better burning and higher energy release during combustion [6].

Oxygen which are the second highest composition of OPF decreased constantly at both holding time. This decreasing trend was also similar to H and N where upon increased of torrefaction temperature, then elemental wt % reduced. Oxygen decreased gradually because oxygen is highly reactive element when exposed to elevated temperature. In addition, oxygen also released as a volatile matter that involved in several chemical reactions to form syngas and other by-products [6].

Decrease in elemental oxygen was due to it being a highly reactive element. During torrefaction, moisture was released as well as formation of CH_4 , C_2H_4 and other hydrogen rich gases. During torrefaction, decreasing of the H and O contents was due to destroyed a hydroxyl group ($-\text{OH}$) in biomass sample and thus producing solid hydrophobic fuel [7].

2) Calorific value analysis

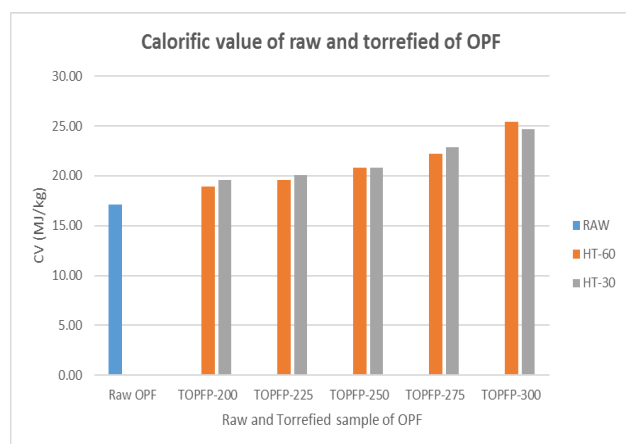


Fig. 2.0 Calorific value of raw and torrefied OPF at HT-60 and HT-30

From Figure 2.0, the calorific value for torrefied sample with heating time of 30 and 60 minutes increased gradually. Calorific value is dependent on the chemical energy that is stored in the sample which is called as energy density. As energy densities increase the calorific value increases [8]. Other than that, higher carbon content as shown in Fig. 1.0 (a) and Fig. 1.0 (b) led to the higher calorific value as more carbon-carbon bond with higher

energy content existed as compared to carbon-hydrogen and carbon-oxygen bond with weaker energy bond [9].

3) Proximate analysis

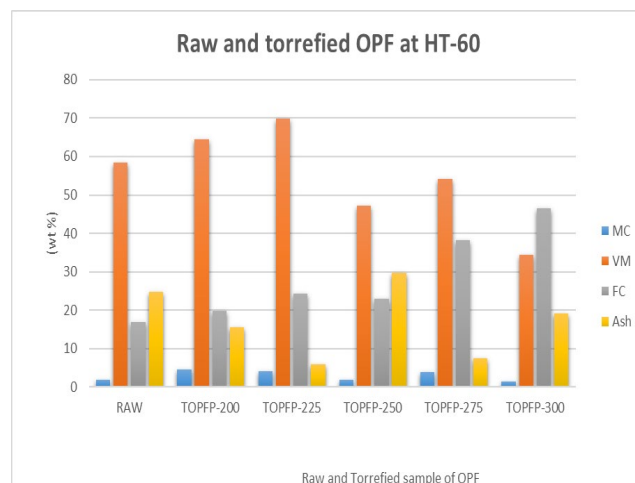


Fig. 3.0 (a) Proximate analysis of raw and torrefied OPF at HT-60.

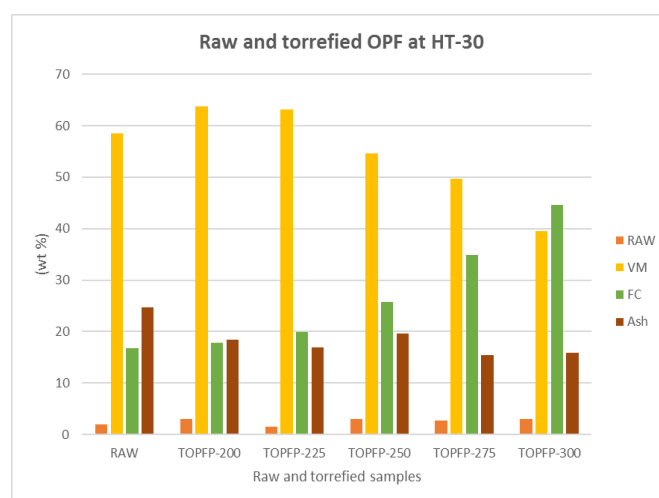


Fig. 3.0 (b) Proximate analysis of raw and torrefied OPF at HT-30.

Fig. 3.0 (a) and Fig 3.0 (b) shows the proximate analysis for raw and torrefied OPF where the wt% of VM, FC, MC and ash content were displayed. The raw OPF in Fig. 3.0 (a) or Fig. 3.0 (b) contained higher volatile matter followed by ash, fixed carbon and moisture content and the proximate analysis of torrefied OPF at holding times of 60 and 30 minutes. In Fig. 3.0 (a), it shows that moisture content decreased inconsistently from 4.61 wt% to 1.40 wt%, which is similar to the volatile matter that decreased inconsistently from 64.57 wt% to 34.33 wt%. Meanwhile, the fixed carbon and ash increased inconsistently which are 19.92% to 46.55% and 15.51% to 19.12% respectively.

However, in Fig. 3.0 (b), overall the moisture content and volatile matter decreased as the temperature increased while the fixed carbon and ash increased inconsistently. Decrease in volatile matter was predicted in order to produce combustion efficiency and thus release lower gaseous pollutants. In addition, the volatile matter decrease also due to the variance in cellulosic content which is carbohydrate as it is the main fraction in lignocellulosic biomass. At the same time, they are easily degraded during thermal treatment [7].

For fixed carbon, the values were increased due to the removal of moisture content and volatile matter [10]. High fixed carbon is necessary to produce a good quality biochar. Lastly, ash content decreased with heating time as an effect of concentration [2]. This is corresponding to the experimental result as the ash content is 18.38 % at 200°C and gradually decreased to 15.83% at 300°C. The ash content of raw sample is found to be greater than torrefied.

Low amount of ash is good as it will reduce ignition and combustion problems such as fouling corrosion, sintering and low ignition time of biomass fuel [11].

B. Biomass composition of oil palm frond (OPF)

Fibrous composition of OPF in terms of hemicellulose, cellulose and lignin were determined via Technical Association of Pulp and Paper (TAPPI) method and the results summary of each composition are displayed in Table 4.4. It was found that the highest percentage of the composition is hemicellulose followed by cellulose and lignin. These results are agreeable to the fibrous composition range reported from previous studies as shown in Table 4.2.

1) Determination of lignin

Table 2.0 Determination of lignin

Ao	Ae	Percentage of lignin (%)
-0.0023	0.0036	21.00

Ao = Value absorbent of blank solution
Ae = Value absorbent of sample in mixture

For lignin, it is a very important component in biomass as it provides plant tissue and individual fibers. The strength and stiffening of the cell wall give high protection towards carbohydrate from damaging the compound chemically or physically [12]. Researchers also reported that lignin is the most complex structure among the three compound in biomass due it high molecular weight and insoluble criteria. At typical biomass pyrolysis with temperature of 300 to 500 °C, lignin is hardly degrading, thus, lignin is better to be pretreated chemically compared to physically.

The lignin degradation technique that has been used in this experiment was to pretreat the sample of OPF by adding the mixture of potassium permanganate (KMnO₄) to sulfuric acid (H₂SO₄) with volumetric with ratio of 4:1. The use of diluted sulfuric acid is to dissolve most of the hemicellulose as well as increasing the susceptibility of cellulose but breaking less lignin of OPF [9].

As shown in Table 2.0, a UV-vis test was applied to determine the absorbent value of blank and mixture solution first. However, during the UV-vis test, the reading of absorbent for the first and second time of mixture was too high. Higher reading of absorbent was due to the high concentration of mixture. Therefore, further dilution was being done to the high concentration of mixture. After the results of blank and mixture absorbent were obtained, the Kappa number is determined by using a formula of Eq(1). Then, the percentage of lignin is obtained by using the kappa number into the following formula Eq(2).

Experiment was repeated for three times and the results are compared with theoretical range reported in previous literatures for OPF. The closest reading was chosen with lignin composition of 21 wt%. It also shown that, the lignin content is the lowest composition among the lignocellulosic composition as compared to hemicellulose and cellulose.

2) Determination of cellulose

Table 3.0 determination of cellulose in OPF

Initial weight of sample (g)	2.042
Weight of filter paper (g)	1.070
Weight after of sample and filter paper (g)	2.210
Final weight of sample (g)	1.140
Percentage of cellulose (wt%)	44.17

For the determination of other lignocellulosic composition which are hemicellulose and cellulose, the holocellulose need to be prepared first. Holocellulose is the combination of hemicellulose and cellulose. The holocellulose was prepared by mixing the OPF

sample with distilled water and treated with acetic acid and sodium chlorite. Sodium hydroxide solution was used as the cellulose need to be extracted from the hemicellulose [13]. The percentage of cellulose is calculated by using the following formula Eq[3]. Table 3.0 shows the cellulose determination in OPF is 44.17 wt%.

3) Determination of hemicellulose

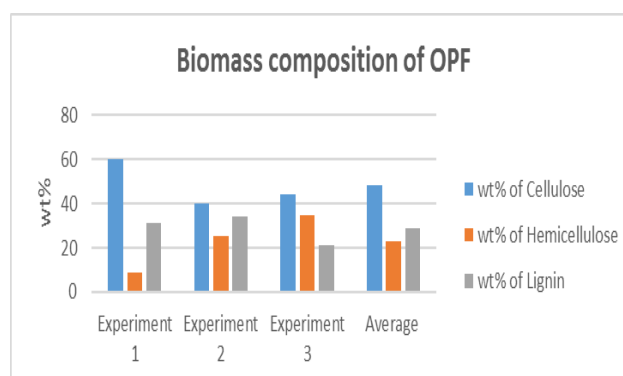


Fig. 4.0 Percentage of biomass composition of OPF

As a conclusion, for OPF fibre composition, by referring to Fig. 4.0 the highest percentage of lignocellulosic is cellulose, followed by hemicellulose and lignin. The third experiment is more accurate as the percentage of lignocellulosic are within the range of fibrous composition reported in literature of Khalil et al. [12] where the percentage of cellulose, hemicellulose and lignin are 40-50 wt%, 34-38 wt% and 20-21 wt% respectively.

According to Zheng et al., [11], large amount decomposition of hemicellulose and cellulose will produce water and light volatile components which can reduce the volatile matter of biochar as shown in Fig. 3.0 (a) and Fig. 3.0 (b). Thus, high quality biochar will be produced of having low volatile matter and higher combustion efficiency and will release lower gaseous pollutants while higher cellulosic composition with lower lignin of OPF has better potential as gasification fuel [10].

4) Determination of extractives.

Table 4.0 Determination of extractive (wt%)

Wi, Weight of sample before extraction (g)	19.50
Weight of (sample + moisture) after extraction (g)	49.20
Wf, Weight of sample after dried (g)	15.55
Extractives (wt%)	20.26

Basically, the extractives in plant fiber could be wax, pectin and even simple sugar. They are easily dissolved in solvent (ethanol-benzene mixture) [13]. There are several indicators that are used as treatments for removal of extractives from surface of OPF; NaOH-OPF, acetic acid-OPF and untreated-OPF. It was also reported that treatment with aqueous glycerol will dissolved more extractives. The extractives of OPF was determined by using the Eq [5].

$$\text{Extractives (wt\%)} = \frac{W_i - W_f}{W_i} \times 100\% \quad \text{Eq(5)}$$

5) Summary of biomass composition of oil palm frond (OPF).

Table 5.0 Biomass composition of oil palm frond (OPF)

Component	wt %
Cellulose	44.17
Hemicellulose	34.83

Lignin	21.00
Extractives	20.26

As a conclusion, for raw OPF fibre composition, by referring to Table 5.0, the highest percentage of lignocellulosic component is cellulose (44.17 wt%), followed by hemicellulose (34.83 wt%), lignin (21.00 wt%), and extractives (20.26 wt%).

C. Porosity analysis of raw and torrefied OPF using BET surface area.

For the determination of surface area and porosity of OPF, Brunauer Emmer Teller [BET] method was employed to measure the surface area of oil palm frond. Table 4.3 displays the BET surface area of each raw and torrefied samples in m^2/g . For better data representation, the values of surface area were plotted as shown in Fig 5.0.

Table 6.0 Sample of raw and torrefied OPF

Name of samples	HT (min)	BET surface area (m^2/g)
RS-2	RAW	1.2284
TOPFP-200°C	60	1.3335
TOPFP-225°C		1.4822
TOPFP-250°C		1.3207
TOPFP-275°C		1.8225
TOPFP-300°C		1.8271
TOPFP-200°C	30	1.0683
TOPFP-225°C		1.9605
TOPFP-250°C		0.9000
TOPFP-275°C		1.0124
TOPFP-300°C		1.2232

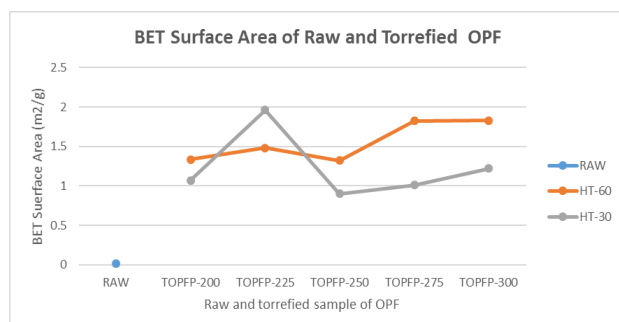


Fig. 5.0 BET Surface Area of raw and torrefied OPF at different HT.

From Table 6.0 and Figure 5.0, generally surface area for torrefied samples of OPF increased at increased torrefaction temperature. The samples were heated with different holding time which was 30 and 60 minutes. From the result shown in the Figure 5.0, the trend of surface area to both torrefied OPF at both holding times is approximately same as the trend of surface area which was upward, downward then upward again.

To explain these three stages trend, initially, the surface area calculated by BET is small as at the initial state when the sample is in raw condition. As the sample undergoes pretreatment, the surface area increases slightly when the temperature increase to 200°C. This is due to rapid precipitation of water and carbon dioxide that occurred on the surface of sample and indirectly caused the surface gaps formed in the sample [11].

When the sample undergoes further increase of temperature until 250 °C, the surface area was decreasing due to a phenomena known as softening. During the softening, only a portion of the surface of sample involved in softening which has organic carbons contained in the biomass [11].

Thus, some part of the gap formed on the surface of sample has been blocked and resulting in the decreasing of surface area. Upon further increase in temperature to 270 °C and 300°C, a significant increase in surface area occurred. During this increase in temperature, the thermal decomposition of some hydrocarbons and volatiles also occurred. The original surface of the sample slowly opens the gaps as volatiles release rapidly at the temperature. In addition, part of each pore on surface of sample has been blocked and caused new pore to form. Consequently, the surface area increases gradually [11].

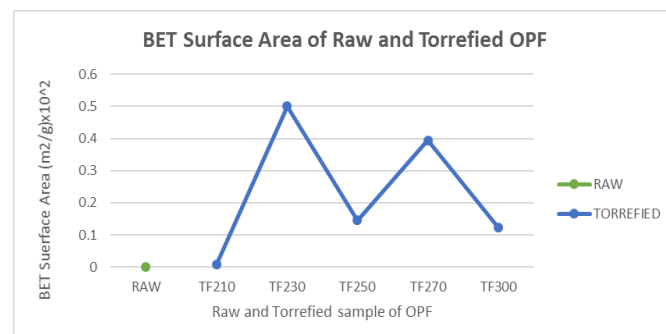


Fig. 6.0 BET surface area of OPF [13]

The data obtained in this study, particularly for torrefied OPF at HT60 minutes is in agreement to past researchers findings. The pattern in changes of pore size increased gradually at temperature 200°C to 230°C, decrease at 250°C, ascending at 275°C and decreasing again at 300°C. However, this pattern was not the same with torrefied at HT30 minutes as it increased at the temperature 300°C.

According to the study by Kristiani et al. [9], it is reported that there are two types of specific surface area of oil palm frond which are external and internal surface areas. For internal surface area, the surface area is smaller than an external area. The specific external area is about 0.6 to 1.6 m^2/g whereas the internal area is about 15 until up to 40 microns. In this study, the use of BET method is only to measure the external specific surface area. The collected data from the experimental result shown that the surface area of OPF is in between the range of 0.6 to 1.6 m^2/g [9].

IV. CONCLUSION

The biomass composition, characteristics of raw/torrefied oil palm frond (OPF) and also other physio-chemical properties were examined. The ultimate analysis was done to evaluate the basic chemical components which will determine the suitability of biomass in OPF as a promising bio-fuel. From this study, it is found that due to the increase in torrefaction temperature, the carbon content increased, meanwhile, oxygen, nitrogen and hydrogen decreased. In addition, the calorific value of OPF also increased gradually. For proximate analysis, as the fixed carbon increased, the volatile matter and moisture decreased. As for biomass composition of OPF, major component is cellulose, followed by hemicellulose, lignin and extractives. The closest experimental data with previously reported data was chosen in which the composition are 44.17 wt%, 34.83 wt% and 21.00 wt%. In addition, the extractives of OPF was also determined and the result is 20.26 wt%. According to Afzanizam [2], high cellulose and low lignin content in biomass composition and also ash composition imply its high potential as bio-fuel. The current study also covers the porosity analysis of raw and torrefied OPF. Due to the increase in torrefaction temperature, the BET surface area will change inconsistently values. But, generally increased as compared to raw OPF.

As a conclusion, the OPF is promising biofuel feedstock when subjected to torrefaction as its has of higher composition of cellulose compared to oil palm trunk and palm leaves. Thus, OPF

also has high potential in renewable energy sources and can possibly replace the utilization of fossil fuel.

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