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Enhanced Oil Palm Waste Properties through Torrefaction and Pelletization

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Abstract. Torrefaction is a thermal pre-treatment process to treat biomass at temperature range of 200-300°C under an inert atmosphere. It was known that torrefaction process strongly depended on the decomposition of the lignocellulosic constituents in oil palm waste namely hemicellulose, cellulose and lignin. The objective of this research is to study the effect of torrefaction on the characteristics of pellet formed from palm mesocarp fiber (PMF), oil palm frond (OPF) and palm kernel shell (PKS) as a solid fuel. The samples were torrefied at 270°C for 30 minutes. The torrefied samples were then densified into pellets through hot press machine at different temperatures (110 °C, 130 °C, 150 °C) and different pressure (10 MPa, 12 MPa, 14 MPa) for 20 minutes to find the optimum pelletization condition. The pellets were analyzed through mass yield, high heating value, fourier-transform infrared spectroscopy (FTIR) analysis, moisture absorption and compression test. Compression test is to study the strength of pellet by identifying the force required to break the pellet. The result shows that the mass yield of PKS was higher than PMF and OPF. The high heating value of torrefied oil palm waste improved as compared to the raw oil palm waste. Through FTIR, the changes of chemical bonds in oil palm waste after torrefaction have been identified. From the analysis of the pellet, the optimum conditions for pelletization were obtained, which are 130 °C and 12 MPa. The moisture absorption rate of torrefied pellet was lesser than raw pellets. Lastly, the torrefaction and pelletization process produced better quality pellets in term of high heating value, durability and more hydrophobic compared to raw pellets which can used as biofuel to replace the coal.

1. Introduction

Oil palm plantations occupy the largest region of agriculture in Malaysia with about 5.4 million hectares of plantation land produce ~ 80% of the biomass making it one of the largest contributors of lignocellulosic biomass in the world [1]. The processing to supply palm oil in advertently produces great quantity of oil palm wastes (OPW) that usually comprise of 13.5% of mesocarp fibre (PMF), 5.5% of palm kernel shells (PKS), and 22% of empty fruit bunches (EFB) in a single ton of fresh fruit bunch

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(EFB) of oil palm [2]. Table 1 shows that oil palm waste have different composition of cellulose, hemicellulose, lignin, ash and extractives [3] [4].

Type of oil palm solid wastes	Chemical components (% dry wt.)					
	Cellulose	Hemicellulose	Lignin	Extractives	Ash	
Empty fruit bunches	38.3	35.3	22.1	2.7	1.6	
Palm kernel shell	20.8	22.7	50.7	4.8	1.0	
Mesocarp fibre	33.9	26.1	27.7	6.9	3.5	
Oil palm trunk	34.5	31.8	25.7	3.7	4.3	
Oil palm frond	30.4	40.4	21.7	1.7	5.8	

Table 1. Oil palm waste composition

OPW is used as a combustion feedstock for power generation in some palm oil mills, which ends up in production of undesirable ash [5,6]. It is thus thought that an alternative approach must be evolved with the aid of transforming OPW into value-added substances inclusive of biochar or activated carbon to enhance the recovery of OPW and divert these wastes from landfill or being a source of air pollution. Since oil palm waste is one of the renewable energy resources, it can be converted to biomass pellet through pelletization process. Pelletization process is a complex interaction between particles and forces. Hardness or durability is one of the important characteristics of pellets and they highly demand in fuel application because of their physical properties and easy in terms of handling [7]. However, in the effort to replace the coal, these raw biomass pellets have some limitations such as high moisture content, low energy yield, low bulk density and poor mechanical properties. Therefore, in order to overcome these issues, torrefaction process can be applied as one of the pre-treatment methods.

Torrefaction is a thermal treatment process at temperature range of 200 – 300 °C where the biomass is heated under inert condition [8]. It's proven that torrefaction method at 270 °C and 30 minutes enhanced the characteristics of biomass in moisture content, mass yield, energy yield and mechanical properties based on previous studies [9,10]. Due to these benefits, torrefaction is chosen as pre-treatment method to upgrade the characteristics of biomass. This research is important because Malaysia is second highest country that produced oil palm waste. So, this research will help palm oil sector to solve the biomass problem turn them into valuable product which is biomass pellet. The objective of this research is to study the optimum condition for pelletization of torrefied oil palm waste and the characteristic of torrefied pellet produced. Torrefaction of oil palm frond, palm kernel shell and oil palm mesocarp fibre were conducted at 270 °C and 30 minutes. Pelletization of torrefied biomass were performed at different temperature (110 °C, 130 °C, 150 °C) and different pressure (10 MPa, 12 MPa and 14 MPa) based on previous studies [11,12]. Several analyses conducted like mass yield, high heating value, fourier-transform infrared spectroscopy, density testing, moisture absorption and pellet strength testing.

2. Experimental

2.1. Materials

The samples used in this study were oil palm frond (OPF), park kernel shell (PKS) and oil palm mesocarp fibre (OPMF). 2 kg of each samples were collected from LKPP Corporation Sdn. Bhd. in Gambang, Kuantan, Pahang. The chemical reagents used were nitrogen gas which is used in torrefaction process and carboxymetylcellulose which is used as binding agent in pelletization process.

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2.2. Pre-treatment of Oil Palm Waste

The collected raw materials were open dried (sun dry or wind) for few days to remove the moisture of the oil palm waste. Then, the raw materials were dried in an oven at 105 °C for 4 hours to provide a basis of tested material. After drying, the raw materials were ground to reduce the size using grinding machine. The raw materials were sieved to size 0.5 - 1.0 mm by using a sieve shaker.

2.3. Torrefaction

A vertical tubular reactor which made from stainless steel with an internal diameter of 22.4 mm and a length of 50 mm used in torrefaction process. About 2 grams of sample was weighed and glass wool inserted first into the reactor before the sample to prevent biomass sample from dropping when nitrogen passing through the reactor. After flushing reactor with nitrogen at constant rate 0.02 L/min for 10 minutes, the reactor temperature was set to 270 °C. After 30 minutes torrefaction, the heater was turned off and reactor is left to cool down to ambient temperature. Then, the torrefied sample collected, weighed and kept in an airtight container.

2.4. Pelletization

The torrefied sample was pelletized by using hot press machine for 20 minutes. At first, the sample was inserted and pre-compressed manually into the mould. The samples within the mould then transferred into Lotus Scientific LS-22025 25 Ton Hot and Cold Moulding Press machine for heating and pelletization purposes. Then, the pressure of 10 MPa and temperature of 110 °C were applied on the pre-compressed sample in mould. The procedures were repeated using different pressures (12 MPa and 14 MPa) and temperatures (130 °C and 150 °C). The author explained in his previous study that he used low pressure and temperature to decrease biomass fuel production energy consumption as well to simplify manufacturing facilities [13]. After the compression process completed, the mould was cooled down to 45 °C to 50 °C then it was removed by using glove. Lastly, the pellets were dried in oven at 50 °C for 30 minutes.

2.5. Characterization of pellet

2.5.1. Mass and Energy yields

The mass yield represents the ratio of mass percentage of torrefied biomass (solid product) left after torrefaction with mass of raw biomass. [14]. The biomass sample was weighed by using weighing balance before and after torrefaction. Energy yield indicates fraction of original energy in the biomass retained after torrefaction and it very important to evaluate the torrefaction performance [15]. The mass and energy yields were calculated by using following equation 1 and 2:

Mass yield(%) =
$$\frac{\text{Mass after torrefaction}}{\text{Mass before torrefaction}} \times 100\%$$
 (1)

$$Energy\ yield(\%) = Mass\ yield \times \frac{\text{High Heating Value of torrefied biomass}}{\text{High Heating Value of raw biomass}} \tag{2}$$

2.5.2. Determination of high heating value

The Parr model bomb calorimeter was used for high heating value measurement of torrefied biomass. The heating value calculated using equation 3:

$$H_g = [(tW - e_1 - e_2 - e_3)/m]$$
 (3)

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where.

t = temperature rise/different

 $W = 2409.26 \text{ cal/} ^{\circ}\text{C}$

e₁ = correction in calories for heat of formation of nitric acid(HNO₃)

 e_2 = correction in calories for heat of formation of sulphuric acid

 e_3 = correction in calories for heat of combustion of fuse wire

= 2.3 x centimetres of fuse wire consumed in firing

m = mass of sample

2.5.3. The Ultimate and Proximate Analysis

The composition of carbon (C), hydrogen (H), nitrogen (N), oxygen (O) and sulphur (S) of oil palm waste conducted by using CHNOS analyser. The raw biomasses were analysed their proximate properties to according to the American Society for Testing and Material (ASTM) standards. The moisture content, volatile matter and ash content are calculated using equation 4 until equation 7 based on ASTM E871 [9], E872-82 [9] and E1755-01 [9] respectively while the fixed carbon content was calculated by using equation 8.

Moisture content (MC%) =
$$\frac{(W_{\text{initial}} - W_{\text{final}})}{(W_{\text{initial}} - W_{\text{crucible}})} \times 100\% = B$$
 (4)

A, Weight loss (%) =
$$\frac{(W_{\text{initial}} - W_{\text{final}})}{(W_{\text{initial}} - W_{\text{crucible}})} \times 100\%$$
 (5)

Volatile matter
$$(VM\%) = A - B$$
 (6)

Ash content (AC%) =
$$\frac{(W_{ash} - W_{final})}{(W_{initial} - W_{crucible})} \times 100\%$$
 (7)

Fixed Carbon (FC%) =
$$100 - (MC + VM + AC)$$
 (8)

2.5.4. Fourier-transform infrared spectroscopy (FTIR)

The structural composition analysis determined the qualitative estimation of hemicellulose, cellulose and lignin content. For this aim, Thermo Nicolet Fourier Transform Infrared Spectrometer used to record FTIR spectra of raw and a few torrefied biomasses. In the region of 500-4000 cm-1, the spectrums were recorded and believed to be accurate within $\pm 1 \text{ cm}$ [16].

2.5.5. Density testing

The density of pellets tested using equation 9 by calculating mass using a laboratory balance and volume calculated by measuring the length and diameter of pellet using vernier caliper.

$$Density = \frac{Mass \text{ of pellet}}{Volume \text{ of pellet}}$$
 (9)

where the formula for volume of pellet is $\pi r^2 h$.

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2.5.6. Pellet Strength

The pellet strength was determined by the tensile crushing strength test, using the SHIMADZU Universal Testing Machine. The torrefied pellet horizontally placed on the stainless-steel base platen, and the load cell of perpendicular applied at a constant crosshead speed of 2 mm/min until the pellet began to crush.

2.5.7. Moisture absorption test

After pelletization, the pellets were put at normal atmosphere. The mass of pellets weighed and recorded every day for five days to determine the moisture absorption of pellets by using equation 10.

Moisture absorption (%) =
$$\frac{\text{Mass of pellets on the day X}}{\text{Mass of pellets on the previous day X}} \times 100\%$$
 (10)

where X represent number of days.

3. Results and Discussion

3.1. Mass and Energy yields

Figure 1 shows the mass and energy yields of palm mesocarp fibre (PMF), oil palm fibre (OPF) and palm kernel shell (PKS). From the figure, the mass yield achieved by PKS (83%) is higher than OPF (75%) and PMF (79%). This is mainly due to lower content of hemicellulose in PKS compared to PMF and OPF [3]. During torrefaction, the high content of hemicellulose in OPF and PMF started to degrade and mass yield become lesser. Niu [17] even mentioned that hemicellulose started degrading at 210 °C. Hence, the mass yield during torrefaction mainly attributed by degradation of hemicellulose. For the energy yield, all the energy yield for the OPF, PMF and PKS were more than 100%. Dirgantara [18] explained that if energy yield of torrefied biomass is greater than 100%, the torrefaction process can improve the energy in biomass. PKS gives higher energy yield because PKS has highest amount of lignin compared to PMF and OPF. From table 1, PKS have 50.7 wt% lignin while PMF and OPF have 27.7 wt% and 21.7 wt% lignin respectively. During torrefaction, most of the hemicellulose degraded and this is the reason the PKS have higher energy yield. From table 2, the PKS has 49.91 wt% carbon content while PMF and OPF have 46.4 wt% and 43.94 wt% carbon content. Hence, higher carbon content resulted in higher heating value in figure 2, contributed to higher energy yield.

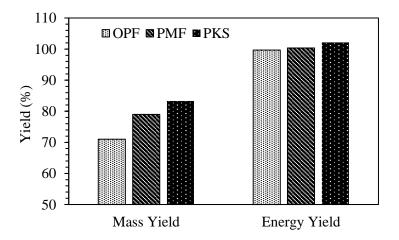


Figure 1. Mass and Energy yield of PMF, OPF and PKS

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3.2. High heating value determination

Figure 2 shows the high heating value of raw and torrefied pellets. The heating value of the torrefied pellets are higher than raw pellets. During torrefaction process, carbonization process occurs that cause conversion from biomass to carbon. This lead to the increase of carbon content and decrease of H/C and O/C ratio in the torrefied biomass to improve the heating value [17]. The heating value of torrefied PKS (23.67 MJ/kg) is higher compared to torrefied PMF (22.45 MJ/kg) and OPF (20.6 MJ/kg). The carbon content in PKS (49.91%) is normally higher than other oil palm waste which give significant heating value compare to others [9].

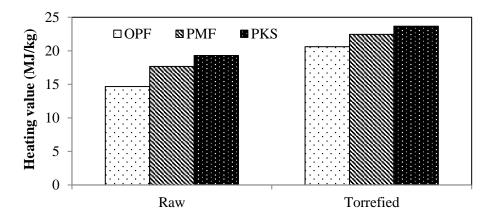


Figure 2. Heating value of raw and torrefied pellets

3.3. The ultimate and proximate analysis

The ultimate and proximate analysis for raw biomass is shown in Table 2 which is obtained from previous studies [8,9,16]. From the table, the fixed carbon of PKS (20.44) was higher than OPF (12.02) and PMF (15.37) which enhanced its heating value as shown in Figure 2. This is mainly due to higher carbon content in PKS which is 49.9% higher than PMF and OPF with 46.4% and 43.94% respectively. The carbon content in PKS mainly contributed by lignin where lignin consists 49.83% of carbon [20]. The oxygen content of raw PMF (50.21%) and OPF (44.88%) were higher than PKS (38.2%), resulting in lower mass yield of PMF and OPF. All three biomasses were not showing big difference in volatile matter. The ash content of PMF was higher than PKS and OPF.

	PKS	PMF	OPF
Ultimate analysis (wt%)			
С	49.91	46.40	43.94
Н	6.94	9.28	6.94
N	3.52	0.39	3.52
0	38.2	50.21	44.88
S	0.72	-	0.72
Proximate analysis (wt%))		
Volatile matter	84.45	84.91	85.10
Fixed carbon	20.44	15.37	12.02
Ash content	3.00	6.10	3.37

Table 2. Ultimate and proximate analysis of biomass

3.4. Fourier-transform infrared spectroscopy (FTIR)

Figure 3 shows the analysis of FTIR to study the chemical functional group for raw and torrefied oil palm waste. Generally, all the spectra to be found different between raw and torrefied oil palm waste. In the region 1, the spectra shown with the band of 3000-3600 cm⁻¹ corresponds to the vibrations of hydroxyl -OH which mainly found in lignin, cellulose and hemicellulose [21]. The intensity at this band for torrefied OPF, PKS and PMF is much broader than raw samples. This clearly show that during torrefaction the hydroxyl group which bond with the oil palm waste was breakdown and removed. In the region 2, the band 2800-3000 cm⁻¹ corresponds to the vibrations of saturated aliphatic C-H with methylene group in cellulose [22]. This band clearly can be seen in raw oil palm waste compare to the torrefied oil due to certain amount of the cellulose decompose during torrefaction. There was sudden decrease in the intensity at region 3 with band 1741 cm⁻¹ related to unconjugated C=O valence vibration of xylan which present in hemicellulose [23]. The torrefied OPF, PKS and PMF shows small peak at this region compare to raw biomass due to degradation of hemicellulose. In the region 4, small intensity peak were shown by torrefied PKS, PMF and OPF which related with benzene and aromatic rings in lignin [24]. The torrefied PKS shows strong peak compare to PMF and OPF which show its high content of lignin. The region 5 shows the intensity of C-O stretching and C-O deformation related with C-OH (ethanol) [25]. The torrefied OPF has strong peak followed by torrefied PMF and others.

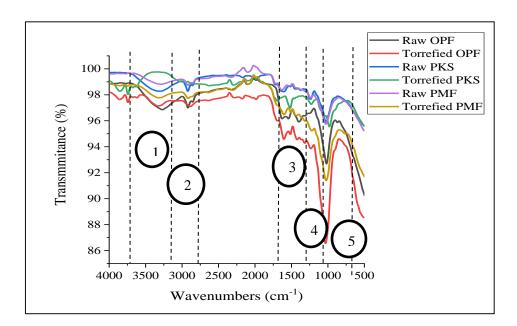


Figure 3. Fourier-transform infrared spectroscopy analysis for raw and torrefied oil palm waste

3.4. Pellet Density

Figure 4 shows the pellet density of raw and torrefied of PMF, OPF and PKS. Pellet density of torrefied pellets is higher compared to the raw pellets due to its brittle characteristic. After torrefaction, the biomass tends to be more brittle and easier to be compressed in the mould. The PKS pellets have more

pellet density compare to others because of its high lignin content which make it more brittle compare to other biomass [3].

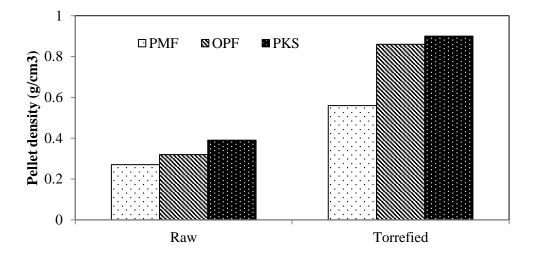


Figure 4. Pellet density of raw and torrefied PMF, OPF and PKS

3.5. Pellet strength

Figure 5 shows the pellet strength of OPF, PMF and PKS. The suitable die temperature and compressed pressure were 130 °C and 12 MPa for these biomasses. The purpose of the tensile strength determination was to study the suitable condition for pellet making. At 110 °C the biomass pellet was could not form due to the insufficient heat supply and at 150 °C the biomass became more brittle due to high temperature and it tend to break easily. Biomass pellets have lower strength at 10 MPa and 14 MPa because at it doesn't get enough compression force at 10 MPa whereas at 14 MPa it is over compressed and make the pellet not strong enough.

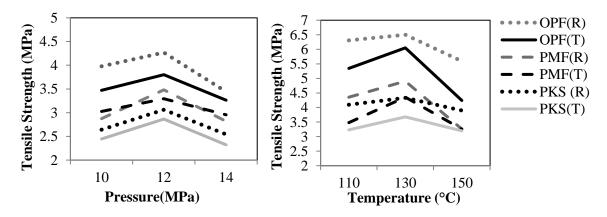


Figure 5. Raw and torrefied pellet strength at different a) pressure and b) temperature

3.6. Moisture absorption rate

Figure 6 shows the moisture absorption rate of raw and torrefied pellets in 5 days. The moisture absorption rate for torrefied biomass is lower than raw biomass due to degradation of hemicellulose [9]. Olugbade [26] explained that during torrefaction, degradation of hemicellulose causes loss of hydroxyl group which is important component in biomass for binding of moisture from surrounding. When temperature increases, more hemicellulose started to degrade, and more hydroxyl group purge out by devolatilization which make the biomass behave hydrophobic and it absorb less moisture.

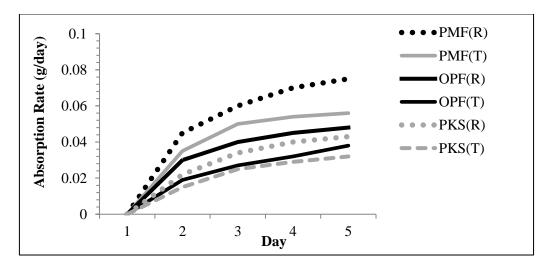


Figure 6. Moisture absorption rate in 5 days for torrefied and raw pellets

4. Conclusions

Torrefied biomass pellets show upgraded properties as compared to the raw biomass pellet. The result shows that torrefied pellets have high heating value compared to raw pellets. The fourier-transform infrared spectroscopy (FTIR) analysis clearly shows that there were changes in functional groups between raw and torrefied oil palm waste. It is proven that to make best quality pellet, the optimum conditions of pelletization are at 130 °C and 12MPa. Lastly, the moisture absorption rate for torrefied pellets are lesser compared to raw pellets. These torrefied pellets by torrefaction and pelletization process have some benefits such has better high heating value, low moisture absorption and better pellet strength compared to raw pellets. Hence, these torrefied pellets have potential to be used as substitute for coal in combustion units, thermal power plants and electricity generation.

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