

PELLETIZING PROPERTIES OF RAW AND TORRIFIED EMPTY FRUIT BUNCHES (EFB)

by

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DECLARATION

I declare that this thesis entitled "Pelletizing properties of raw and torrified empty fruit bunches (EFB)" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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Pelletizing properties of raw and torrified empty fruit bunches (EFB)

ABSTRACT

This study research focused on the properties of raw and torrified pellet from EFB by varying different parameters, including energy consumption, moisture adsorption, meyer hardness test and pellet density. To save storage space and transport costs, it can be compressed into fuel pellets of high physical and energetic density. The pelletizing properties were determined using single pellet press and pellet stability was determined by compression testing. Pelletization of raw and torrified empty fruit bunch (EFB) from compression single pellet press (SPP) was investigated to quantify the energy consumption and pellet properties, including moisture adsorption, pellet density and Meyer hardness . Energy consumption for torrified EFB pellet were significantly higher than those for raw EFB pellet, while the moisture adsorption rate of torrified EFB pellets increased with increasing the severity of torrefaction. Decomposition of hemicellulose and lignin reduced the plasticity of raw sample contributes to increase energy consumption during compression. The hardness of pellet made at same pelleting condition decreased with the increasing of size of sample. The densities of torrified EFB pellets were higher than raw EFB pellet due to the loss of chemically bonded water and low melting point compounds during torrefaction which act as binding agent. The properties of pellets were affected by the removal of most low melting or softening point components.

Sifat-sifat Pelet dari bahan mentah dan pengeringan tandan buah kosong

ABSTRAK

Penyelidikan ini memberi tumpuan kepada sifat sifat mentah dan torrified pellet dari EFB dengan keadaan parameter yang berbeza termasuk penggunaan tenaga, penjerapan lembapan, ujian kekerasan Meyer dan kepadatan pelet. Untuk menjimatkan ruang penyimpanan dan kos pengangkutan, ia boleh dimampatkan ke dalam pelet bahan api ketumpatan fizikal dan bertenaga tinggi. Sifat pelletising telah ditentukan mampatan pellet tunggal tekan dan kestabilan pelet ditentukan oleh ujian mampatan. Pelletising bahan mentah dan torrified EFB dari mampatan pelet tunggal tekan (SPP) telah disiasat untuk mengukur penggunaan tenaga dan sifat pelet termasuk penjerapan lembapan, kepadatan pellet dan ujian kekerasan Meyer. Penggunaan tenaga untuk torrified EFB pellet adalah lebih tinggi berbanding EFB pellet dari bahan mentah, manakala kadar penjerapan lembapan pellet EFB terrified meningkat dengan peningkatan suhu torrefaction. Penguraian hemiselulosa dan lignin mengurangkan keplastikan sampel mentah menyumbang kepada peningkatan penggunaan tenaga semasa pemampatan. Kekerasan pellet pada keadaan pemampatan yang sama menurun dengan peningkatan saiz sampel. Ketumpatan pellet EFB torrified lebih tinggi daripada pellet EFB mentah kerana kehilangan sebatian air dan takat lebur yang rendah semasa torrefaction dimana bertindak sebagai agen pengikat. Sifat sifat pellet terjejas oleh pembuangan takat lebur rendah atau komponen titik lemah.

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Energy has always played an important role in our life, survival, and for the development of our civilization. Biomass was one of the major energy sources since the beginning of human population, and still play an important role in develops country economic. However, biomass has become worldwide attention because due to global climate changes caused by increased non-renewable resource consumption. Besides that, rapid progress economic growth in developing countries, high dependence in global and local transportation on fossils fuel, depletion of sources, pollution, endangered national security of energy importing countries has raised the awareness of the need for non-fossil sources (Demirbas *et al.,*2008).

Malaysia is the largest exporter of oil palm in the international market. In recent years, growing attention worldwide has focused on the use of ligno-cellulosic biomass residues as a feedstock to produce biofuel pellet as an alternative to fossil fuel. Ligno-cellulosic biomass residues plays a major role in the production of sustainable energy, because it is abundant, relatively inexpensive and often locally available (Byeong-Ill Naa *et al.,* 2013). In fact, Malaysia is the one of the main producers of palm oil in the world. Oil palm industrial waste, 53% of biomass is made up from Empty Fruit Bunch (EFB) and other 32% and 15% are made up from mesocarp and palm kernel shell respectively. The producing of ash, which is later used as fertilizer from EFB in the past has been stopped. Due to environmental issue. EFB has been recycled nowadays to avoid this issue (A.S Baharuddin *et al*., 2009). Their

composition has a great effect on pellet quality and the pelletizing process. Biomass pellets are produced in pellets mills by pressing the biomass through cylindrical shaped press channels in which the biomass is exposed to high pressure and heat that arises from the high friction between the biomass and the press channel walls (Wolfgag Stelte *et al*., 2011).

Torrefaction is a pre-treatment process where biomass is heated in the absence of oxygen at low heating rates which is less than 50°C /min to temperatures between 200°C and 300°C to produce a darkened material with improved chemical and physical properties. The moisture and oxygen-rich volatile materials with low calorific value are driven off will result in a reduction in hydroxyl groups that can form hydrogen bonds with moisture and a greater mass loss to energy loss producing a more energy dense fuel during torrefaction. This will ascribed to decomposition of the hemicellulose fraction, which binds the cellulose fibrils in the cell wall providing structural integrity. The process often requires drying of the fuel prior to torrefaction, the process is overall endothermic and so requires an energy input while torrefaction improves the chemical and physical properties of raw biomass. Some energy will be lost in volatile materials evolved which can be combusted to provide some of heat requirement of process during torrefaction (Mcnamee *et al.,* 2016).

1.2 Problem Statement

Malaysia's palm oil industry leaves behind huge amount of biomass from its plantation and milling activity. These large values of biomass surely need a huge budget to form into useful product. The availability of new technology are really required to convert this sustainable source of palm oil into various types of value added products that will generate money and also save the cost of treatment (Shuit *et al*., 2009). Biomass is uneven, fluffy, dusty in its raw form and these characteristics make it difficult to store, transport and utilize. Pelletization of biomass can be easy handling, reduces transportation and shorter pellets are easier to handle. Although densification of biomass through pelletization can improves some characteristics, there might be some problem during processing which can create pellet breakage and the production of fines or dust (Andrew Duncan *et al.,* 2013). This study intent to produce pellet from EFB to reduce the quantity oil palm waste. In order to increase the value products of energy densification and easy to be handling by converting this biomass into pellet.

1.3 Objectives

The main objectives are to investigate the physical properties of raw and torrified pellet of EFB that derived from oil palm waste. Below are the specific objectives regarding this study:

- i. To produce the pellet from raw and torrified EFB.
- ii. To characterize the raw and torrified properties of EFB pellets.

1.4 Expected Outcome

This study is focusing on pelletizing process onto oil palm wastes. The consideration that need to ensure is the physical properties of raw and torrified pellet of EFB. The density, energy consumption, hardness test and water adsorption and permeability, compression ratio and particle size of raw and torrified pellet will be investigate. Each pellet gives different effect when load, pressure and energy is being applied onto it. The pellet will be produced using Universal Testing Machine (UTM) with compression jig.

CHAPTER 2

LITERATURE REVIEW

2.1 Renewable Energy

Energy can be divided into two types, non-renewable energy resources and renewable energy resources. Non-renewable energy resources such as coal, oil and natural gas. Coal is formed from fossilized plants and consisting of carbon with various organic and some inorganic compound. Oil is a carbon-based liquid formed from fossilized animals. Oil is widely used in transport and industry. Natural gas is methane and some other gases trapped between the seams of rock under the earth's surface. Renewable energy resources such as solar, wind, tidal, wave, geothermal, hydrological and biomass. Renewable energy sources quickly replenish themselves and can be used again. Therefore, for this reason, non-fossil fuels are sometimes called infinite energy resources.

In the context of biomass for energy, it is often mean the plant based material which is derived from the reaction between carbon dioxide $CO₂$ in the air, water and sunlight, through photosynthesis to produce glucose that form the building blocks of the biomass. In addition, the energy stored in the chemical bonds of the biomass has been exploited by burning it as a fuel. One important factor considering the use of biomass as energy sources is to reduce the global warming issue since burning new biomass contributes less $CO₂$ to the atmosphere, because replanting biomass ensures that the $CO₂$ is absorbed and returned for a cycle of new growth (P. McKendry *et al.*, 2002).

2.2 Biomass

In Malaysia, it can be considered that this country is one of the largest producer of biomass. It is because the palm oil industry in Malaysia had contribute 85.5% of the total biomass hence offering the great potential for large-scale power generation (Umar *et al.,* 2013). Biomass is organic materials that could be one of the promising renewable energy sources and could be utilized as solid, liquid and gas fuels (Uemura *et al.,* 2011).

Biomass is the amount of living organism in certain habitat or derived from the living organism itself. Usually it is whether from plant based material both comes from animal or plants. Biomass can be categorized into four groups namely agricultural waste, wood residues, energy crops and also municipal solid waste (Aziz *et al.,* 2011). Biomass had already become one of the most regularly used as a renewable sources of energy few decades ago. Due to the huge amount of biomass generated from the oil palm industry, it will be a waste if the biomass is not utilized properly (Sabil *et al.,* 2013).

In this study, we are focusing on agricultural waste to convert it to energy. The agricultural waste is a production of waste from agricultural operations. It is derived whether from farms, plantation, and also harvest waste. All of these could be convert to become renewable energy. There were many challenges to utilize the raw biomass due to its characteristic. This is because the raw biomass is usually high in moisture content, low energy density, difficult to store and poor grind ability (Aziz *et al.,* 2011). Biomass is constantly formed from the interaction of $CO₂$, air, water, soil and also sunlight. It is also undergoing the process of photosynthesis. Biomass is a sustainable source for generation of low carbon energy and liquid fuels and it is expected to play a critical role in the future.

(Woei L. Saw & Shusheng Pan,2012). It also is a bulky and inhomogeneous material that make it difficult to comminute into small particle and has relatively low energy density and high moisture content (Wolfgag Stelte *et al.,* 2011).

2.2.1 Biomass from Empty Fruit Bunch (EFB)

Malaysia is the one of the main producers of palm oil in the world. Oil palm industrial waste, 53% of biomass is made up from Empty Fruit Bunch (EFB) and other 32% and 15% are made up from mesocarp and palm kernel shell respectively. The producing of ash, which is later used as fertilizer from EFB in the past has been stopped. This is because of environmental issue. EFB has been recycled nowadays to avoid this issue (A.S Baharuddin *et al*., 2009). Its amount were accounted about 15.8 million ton per year and it will be the huge amount for production sustainable resources that derived from oil palm fresh fruit bunches (Ying, Keat, Nadiah, Abdullah, & Cheu, 2014). Table 2.1 presents palm oils residuals in Malaysia and Indonesia.

There are many types of oil palm biomass. For example, fronds, trunk, shells, fibre, palm oil mill effluent (POME) and empty fruit bunches. Fronds is comes from leaves of oil palm tree. Trunk is available at the end of plantation life cycle. Shells remains after palm kernel oil extraction. Fibre remains after crude palm oil (CPO) extraction from fruit bunches. POME is a liquid by-product from sterilization and milling process. EFB remains after removal of oil palm fruits. This has been showed in Figure 2.1.

 Figure 2.1: Types of oil palm biomass (Source: Agensi Inovasi Malaysia)

2.3 Torrefaction

Generally, torrefaction can be defined as mild pyrolysis of biomass at temperatures between 200°C and 300°C, normally in absence of oxygen (OngWei Wang *et al*., 2013). Torrefaction also is the one process of thermal pretreatment before combustion and gasification. It helps removes water content and low molecular weight organic volatiles and decompose long polysaccharides chains of biomass materials (M.J Wang *et al.,* 2011). A hemicellulose is partially decomposed and some low calorific volatiles are released from the raw biomass and also enhancing the remaining biomass energetic potential under these conditions. It is possible to obtain new biomass fuels, whose properties range between biomass and coal in this way (Costa & Costa, 2015).

The torrefaction process has been used for high energy density of lignocellulosic biomass. Torrified biomass should be evaluated in terms of energy yield. Thus, it also provides suitable chemical and physical characteristics for long distance transportation and long term storage. Torrefaction of biomass accompanies weight loss depends on torrefaction conditions. During torrefaction of biomass, most volatile compounds are removed from biomass vapors and result in a higher energy density. In addition, the advantages of torrefaction includes higher calorific value or energy density, lower moisture content and better grinding (Byeong-Ill Na *et al.,* 2013). The low power (100-200W) was insufficient for torrefaction power for heating process due to lower heating value (Huang et al., 2016).

2.4 Microwave reactor

Microwave is an electromagnetic wave that comes with wavelength between the infrared light and radio waves. On the other words, microwaves are electromagnetic waves with frequencies between 300 MHz and 300 GHz, and thus the corresponding wavelengths are between 1m and 1mm, respectively (Huang *et al.,* 2016). Based on previous study, microwave also was defined as lie between infrared and radio frequencies in electromagnetic spectrum and the frequencies that commonly used is 2.45GHz (Y. Huang et al., 2016). Microwave heating is a selective, rapid, uniform and energy, saving method without direct contacts with heated materials. Thus, it also has many potential advantages in processing materials. However, not all materials can be heated using microwaves (M.J Wang *et al.,* 2011). There are three types of interaction between materials and microwave irradiation, which are reflective (conductors), transparent (insulators) and absorptive (dielectrics) reactions (M.J Wang *et al.,* 2011). Microwave heating includes two mechanisms: dipole rotation and ionic conduction which both of these mechanism is able to heat materials quickly and uniformly. Heating rate will be defined as heat that was generated by migration of ionic species or rotation of dipolar species or both which is can absorb the materials. On the other words, heating rate also called as dielectric heating (Y. Huang *et al*., 2016). Based on previous study, the increasing of power level will gives the higher heating rate (Huang *et al.,* 2016).

2.4.1 Performance of microwave

Microwave heating has been used for the pyrolysis or torrefaction (mild pyrolysis) of various biomass feedstocks, such as scrap tire, wood, sewage sludge, coffee hulls, oil palm biomass, microalgae, sugarcane bagasse, wheat straw, and corn stover (Huang *et al.,* 2016). Besides that, microwaves produces heat by converting electromagnetic energy to thermal energy instead of heat transferring. Only specified materials that have dielectrics properties can be heated by microwave energy delivered directly to materials through molecular interaction within the electromagnetic field (M.J Wang *et al.,* 2011). Usually, the typical microwave reactors work at a frequency of 2.45GHz. However, as for microwave reactor, it is different from usual microwave because of the condition of heating itself. The influence of microwave power level on both maximum temperature and heating rate was substantial (Huang *et al.,* 2016)

2.5 Pelletizing

Torrified sawdust was then compressed into pellets in a single die compression test unit. In combination with pelletization, torrefaction aids in addressing the logistics issue for untreated pellets such as low energy density and low hydrophobicity. The making of torrified pellets and properties of both torrified sawdust and pellets such as energy consumption during compression of torrified pellets, pellet density and moisture content have been measured to identify suitable torrefaction and densifications conditions. Torrified pellets can potentially reduce handling costs and result in a solid fuel of standardized shape and size that can be fed automatically to solid fuel boilers for heat and power generation (OngWei Wang *et al.,* 2013).

Pelletizing properties of torrified biomass have been analyzed depending on torrefaction conditions (Byeong-Ill Na *et al.,* 2013). The biomass have been milled, dried and mechanically densified during pelletization to enhancing its heating value and burning characteristics (Adrian Pirraglia *et al.,* 2013). Wood pellets are the only solid biofuels which have global market and their energy density can be increased by torrefaction process. Besides that, it can lower the pellets mechanical properties like low dust formation is desired when producing pellets during torrefaction process. It also can reduces the problems during transportation and handling like easily to handle (Lei Shang *et al.,* 2012). Biomass pellets are produced in pellets mills by pressing the biomass through cylindrical shaped press channels in which the biomass is exposed to high pressure and heat that arises from the high friction between the biomass and the press channel walls. As a result, it will be easy to comminute into small particle and can save storage space and transportation costs. Their properties were determined using a single pellet press and press stability was determined by compression testing (Wolfgag Stelte *et al.,* 2011).

2.5.1 Pellet Characteristics

2.5.1.1 Hardness test (Meyer hardness)

The Meyer hardness of prepared pellets should be obtained from the stress–strain curve from the crushing tests. The Meyer hardness of torrefied pellets should be lower than control pellet under same temperature and compression test. This is because of the amount of available hydrogen bonding sites is reduced during torrefaction. So that the strength of torrefied pellets is lowered compared to pellets made from untreated sawdust. Apart from

that, the moisture content of the torrefied sawdust is lower, which can be increase in the glass transition temperatures of the remaining hemicelluloses and lignin (C.Wang *et al.,* 2013). Hardness is one of the important characteristics of commercial pellets, which reflects the strength of individual pellet. Forces that cause damage to pellets during handling, transportation, and storage may be divided into compression, impact, and shear forces. The Meyer hardness against compression from a static indentation test was measured. When a force is applied to the pellet, the pellet will be deformed. The Meyer hardness, *Hm*, defined as the static indentation hardness, should be determined from the applied force, probe diameter and indentation depth. Meyer hardness reflects the resistance of the solid object to various kinds of permanent shape changes when an axial force is applied. The Meyer hardness should be calculated from (Equation 2.1) that was develop by (Hui Li *et al.,*2012).

$$
H_m = \mathbf{F} / \left[\pi \left(Dh - h^2 \right) \right] \ (2.1)
$$

Where

 H_m = Meyer hardness $h =$ Indentation depth $F =$ Force $D =$ diameter of rod

2.5.1.2 Energy consumption

There are two energy consumption processes associated with pellet preparation. One is the compression of sawdust for pelletization, which accounts for the major energy consumption, and the other is from the extrusion of the pellets out of the die, with a much less energy being consumed (C. Wang *et al.,* 2013). Forces and displacement to form pellets were recorded to calculate the energy consumption. The energy consumption associated with compaction and extrusion were obtained by integrating the curve. Energy consumption are specifically referred to as the mechanical energy for compaction and extrusion. The decomposition of hemicellulose and lignin reduced the plasticity of sawdust, which then contributes to the increased energy consumption during the compression and extrusion (Hui Li *et al.,* 2012).

2.5.1.3 Pellet density

An important parameter, which determines the energy density per volume and the hardness of the pellets can be defined as pellet density. The single pellet density should be calculated from measured mass and volume of individual pellet from (Equation 2.2). The higher compaction pressure or higher die temperature is required to increase the density of torrified pellets. The reason why decreasing of pellet density is mainly related to the loss of chemically bonded water and low-melting point compounds which act as a binding agent when softened at 100°C . Higher die temperature is thus needed to make rigid torrefied pellets (Hui Li *et al.,* 2012). All torrefied biomass pellets have a similar density, however after moisture absorption, the pellet density decreased from about 1g to 0.7g for torrefied

pellets. The formation of more inter-particle gaps and voids by torrefaction is the effect of why the density of torrefied pellets is about 10% lower than raw pellet before moisture absorption (C. Wang *et al.,* 2013). The strength of pellets was measured 48h after production. Before the strength measurement, dimensions (length and diameter) and mass of each pellet were measured to calculate pellet density. The lower particle size will gives the higher density for all biomass (Mo *et al.,* 2014). Besides several other material and process parameters, particle size plays a major role in pellet quality and density. It significantly contributes to the mechanical strength of biomass pellets (Harun & Afzal, 2016). In the past research (Hui Li *et al.,* 2012), the higher compaction pressure or higher die temperature is required to increase the density of torrified pellets.

$$
o = \frac{m}{V}, \quad (2.2)
$$

where

 ρ = density,

 $m =$ mass

 $V =$ volume

2.5.1.4 Moisture adsorption and permeability

Moisture adsorption is an important parameter when considering pellet storage and the increasing the moisture content above the optimum has a negative effect on the pellet density. The molecular mobility of amorphous polymers (lignin and hemicelluloses) and

other low glass transition temperature extractives also increased related to the increasing of moisture content. Besides that, water molecules also known as plasticizer. However, the mechanical properties of wood also highly sensitive to moisture content. The sample were dried in an oven at 105°C for 24 hours. After that, the sample were placed in climatic chamber at 30°C and 90% of relative humidity. After moisture absorption in climatic chamber, the moisture uptake was measured as the weight difference between the initial dry weight and the equilibrated weight (M. Puig-Arnavat *et al.,* 2016).

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

The materials that was used in this study is the empty fruit bunch (EFB) derived from oil palm wastes. The EFB have been collected from oil palm plantation located at Felda Kemahang, Tanah Merah. The size of the EFB was prepared according to the specific mass and measurements. The physical properties of EFB such as density and the moisture absorption were addressed.

3.2 Methodology

3.2.1 Sample preparation

In this study, sample of raw and torrified EFB were prepared to study a comparison in density, moisture adsorption, Meyer hardness and energy consumption. The preparation for sample of raw, the sample were grinded and sieved at 3 sizes which is $250\mu m$, $500\mu m$ and 750µm while for the preparation of torrified sample, 500µm of EFB was required in this process. Figure 3.1 shows the raw of empty fruit bunch (EFB) that was used in this research.

 Figure 3.1 : Raw of Empty Fruit Bunch (EFB)

The raw material samples undergoes several processes before getting through into torrefaction process. After that, it was grinded as shown in Figure 3.2.

Figure 3.2 : Grinded EFB

And then the sample were sieved at 3 sizes which is $250\mu m$, $500\mu m$ and $750\mu m$ based

on Figure 3.3.

Figure 3.3 : Process of sieving EFB

The raw sample weighed of 200g was dried inside the oven for about two hours at 105°C at 24 hours. This method is to remove the whole moisture content from the EFB. The model of microwave used for this process is the reactor microwave with the DAEWOO brand as showed in Figure 3.4. After that, torrefaction temperature of the sample that put inside the microwave were 200°C, 225°C, 250°C, 275°C and 300°C. and then the residence time that setting in this process at 3 time which is 20, 40 and 60 minutes. 30ml/min nitrogen gas was used in this process. The selected power that was used in this process is 100W, 230W, 385W, 540W and 700W. 500micron and 15g of EFB that were required while conducting this process. At this stage, the physical appearance of the EFB will started to show. And then, the effect of heating rate on different power, effect temperature on power selection, effect magnetron on power selection, effect power on fractional torrefied biomass and effect temperature on mass loss was recorded to choose the best selected power. The main reason to choose the best selected power because of the best power will produced the best torrefied material. Besides that, it also to proven either this biomass can be used as renewable energy.

Figure 3.4: Reactor Microwave

The process of this study was planned according to the flow of the process. The pressing mold the 2D drawing schematic diagram flow of pellet mold has drawn using Microsoft Visio. The schematic diagram of pellet mold based on Figure 3.5.

 Figure 3.5: Schematic diagram of pellet mold

3.2.2 Sample preparation of pellet from raw and torrefied EFB

For the preparation of pellet from raw EFB, the biomass was loaded in pellet mold, not exceeding 1g per layer which is at 3 different size, $250 \mu m$, $500 \mu m$ and $750 \mu m$ for 0.60g, 0.55g and 0.50g respectively. While for the preparation of pellet from torrefied EFB, 500µm of EFB size was loaded in pellet mold in 0.70g with 10% of glycerol as a binder agent for different temperature and residence time which is 200°C, 250°C and 300°C and 20, 40, 60 minutes. When the all sample was prepared, the process of compression pellet were conducted. The sample was compressed with a maximum pressure of 200 MPa at rate of 5mm/min using an Universal Testing Machine (UTM), equipped with a maximum 5 kN load cell and an electronic data collection system. And then, the pressure was released after 5 seconds, the piston removed and a new amount of sample is load and compress until the pellet had desired dimensions. The sample was pressed by applying a force 1kN-5kN for raw sample and then for torrefied sample 1kN and 5kN. Figure 3.6 shows the UTM that was used in this research.

Figure 3.6: Universal testing machine (M500-50CT)
The value data of force and displacement was recorded to calculate the next testing in this research such as energy consumption and Meyer hardness test.

3.3 Sample Analyses Properties of Pellet

3.3.1 Meyer hardness test

The Meyer hardness against compression from a static indentation test was measured. When a force is applied to the pellet, the pellet will be deformed. The Meyer hardness, *Hm*, defined as the static indentation hardness, was determined from the applied force, probe diameter and indentation depth. Meyer hardness was reflects the resistance of the solid object to various kinds of permanent shape changes when an axial force is applied. The Meyer hardness was calculated using equation (2.1).

3.3.2 Density test

The single pellet density was calculated from measured mass and volume of individual pellet as shown in equation (2.2). The strength of pellets was measured 24h after production. Before the strength measurement, dimensions (length and diameter) and mass of each pellet was measured using weighing balance and for the volume was measured by vernier calipers to calculate pellet density. The density, or more precisely, the volumetric mass density, of a substance is its [mass](https://en.wikipedia.org/wiki/Mass) per unit [volume.](https://en.wikipedia.org/wiki/Volume)

3.3.3 Water absorption and permeability test

The sample was dried in an oven at 30°C for 2 hours. After that, the sample was placed in climatic chamber at 30°C and 90% of relative humidity. After moisture absorption in climatic chamber, the moisture uptake was measured as the weight difference between the initial dry weight and the equilibrated weight.

3.3.4 Energy consumption

To calculate the energy consumption, forces and displacement was recorded to form the pellets. The decomposition of hemicellulose and lignin reduced the plasticity of sawdust, contributes to the increased energy consumption during the compression and extrusion.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Result overview

Microwave was defined as electromagnetic wave that comes with wavelength between the infrared light and radio waves. On the other words, microwaves are electromagnetic waves with frequencies between 300 MHz and 300 GHz, and thus the corresponding wavelengths are between 1m and 1mm, respectively (Huang *et al.,* 2016). Based on previous study, microwave also was defined as connectors between infrared and radio frequencies in electromagnetic spectrum and the frequencies that commonly used is 2.45GHz (Y. Huang *et al*., 2016). In this study, the microwave frequencies that was used is similar with Y. Huang *et al.*, study, which is frequencies that used 2.45GHz that assisted to undergoes the torrefaction process for the EFB. This process has to control all the parameter which is power level (watt), temperature (°C), volume of nitrogen (ml/min) and mass of sample (g) during the torrefaction process.

4.2 Effect of torrefaction process

4.2.1 Effect of heating rate on different microwave power.

Table 4.1 shows the heating rate on different microwave power.

 Figure 4.1 shows the results of heating rate effect on different power selection. This results based on same particle size which is 500µm while conducting the sample. Heating rate will be defined as heat that was generated by migration of ionic species or rotation of dipolar species or both which is can absorb the materials. In other words, heating rate also called as dielectric heating. Based on previous study, the increasing of power level will gives the higher heating rate (Huang *et al.,* 2016). The trend in Fig. 4.1 shows the same with previous study which is the higher power level contributes to higher heating rate. The highest power level produced the highest heating rate that is 700 W at 41.60 °C/min.

Figure 4.1: Heating rate effect on power selection.

4.2.2 Effect of temperature profile on different power

For starting the handling torrefaction process, 500µm of EFB were heated in microwave. The power level that was used in this process which is 100 W, 230 W, 385 W, 540 W and 700 W. The heating process was run in 40 minutes for maintaining its inert atmosphere. 30 ml/ min of nitrogen was purged inside the microwave. Based on Fig. 4.2, the best selected power level were chosen.

At higher power such as 385W, 540 W and 700 W, the trend shows the slow temperature rising at minutes 1 until 10, followed by rapid temperature rising and lastly reduction state or cooling state. Besides that, the trend of the temperature at low power which is 100 W and 230 W only experience two steps that are slow temperature rising then followed with temperature-reduction state. Based on previous research by Huang *et al*, which stated that at low power such as from 100W to 200W was insufficient for torrefaction power for heating process due to lower heating value. The heating rate for 100 W and 238 W was 2.86 and 4.54 °C/min.

As a conclusion, the most suitable power to be selected in this study was 385 W because they were achieve all the criteria need in this study like achieve the optimum temperature within three stage of heating rate.

Figure 4.2: Temperature profile in different microwave power selections

Based on Fig. 4.3, the trend shows the higher power level used, the higher electron will be produce in the microwave. During heating process, the on/off light in the microwave shows about the formation of electron inside the microwave. The on/off peak inside the microwave quit stable at power level 100W, The peak 'on' are delay for 1 mins in this power. The electron will be produce more at 100 W but this power cannot be chosen as a best power due the low heating rate. The peak has too sharp frequency and their peak 'on' only delay for a few second only at 200 W. The electron will produce in this power are too low and not suitable as a selected power in this situation. The peak 'on' delay for a long time at high power such as 540 and 700 W especially for 700 W that are delay for 5 minutes. At 385 W, the peak 'on' delayed for 1-2 minutes. So, as a conclusion this power that can be selected as a best power.

4.2.4 Effect of percentage of fractional torrefied biomass on different power

The comparison yield of solid, liquid and gas after heating process in different microwave power was shown in Figure 4.4. The minimum and maximum power was used for this study was 100 W and 700 W. The power use was influenced the final percentage of the solid, liquid and gas. At 100 W, the remaining solid produce after the heating process was with 78 % which is the highest percentage in this trend. Whereas at 700W, the remaining solid produce after the heating process was with 22% which is the lowest solid percentage in this trend.

The trend for remaining liquid percentage was increased due to increasing of power, different from the trend percentage of solid. The percentage of remaining liquid was 7% at 100 W whereas there are slightly increased to 20% at 700 W when applied power towards EFB sample.

The condensable product increased after the torrefaction process with increasing of power and torrefaction temperature used towards the sample. Besides that, the increasing of power used from 100 W until 700 W, so the trend of percentage remaining of gas was increased. The result obtained from this study was at 700 W, the percentage of remaining gas was 50% which is the highest percentage where at 100 W, the percentage of remaining gas was 10% which is the lowest percentage.

Figure 4.4: Effect rate of heating power selection on fractional torrefied biomass.

4.2.5 Effect of torrefaction temperature on biomass mass loss

The highest of the weight loss obtained for EFB was about 13.90% at 300°C in minutes 60 and the lowest mass loss was at 200 \degree C in minutes 20 that are 6.2% was shown in Fig. 4.5. This was expected because as time goes by, more water in EFB is evaporated leaving behind the solid and lead to the mass loss.

As a conclusion that can be concluded after finished all the study about effect of heating rate on different power, effect temperature on power selection, effect magnetron on power selection, effect power on fractional torrefied biomass and effect temperature on mass loss, the best power that comes out with the high amount of torrefaction product was 385 W and then for the mass had been selected in this study was 15 g. This mass was already proved as suitable mass because the process selection will come out with maximum yield among the other mass.

Figure 4.5 : Effect of torrefaction temperature on biomass mass loss

4.3 Appearance study of raw and torrefied EFB pellets

Table 4.2 shows the appearance of raw pellet while the appearance of torrefied pellet was shown in Table 4.3. The pressure force that have been used for the raw pellet, 1kN until 5kN while for the torrefied pellet, only 1kN and 5kN. For pelletizing torrefied pellet, glycerol have been used and also act as binder agent.

SAMPLE	PELLET				
	1kN	2kN	3kN	4kN	5kN
500mic					

Table 4.2 : The appearance of raw EFB pellets on different force

Table 4.3 : The appearance of torrefied EFB pellets on different force

4.4 Pellet compression study

The compression diagrams for pelletization pellet as a sample dependence of force (N) on deflection (mm) are presented in Fig. 4.6. The compression testing is commonly related between pelletization force and deflection. Deflection increased with increasing the pelletization force. The highest pelletization force is 50485N with the deflection 21.391mm

which is at 3kN, while the lowest pelletization force is 24053 with the deflection 15.722mm which is at 5kN.

Figure 4.6 : Typical force vs deflection of EFB pellet compression

4.4.1 Effect of density of raw and torrefied EFB pellets

Pellet density is an important parameter, which determines the energy density per volume and the hardness of the pellets. In this study, the density of single pellet was calculated based on the measured pellet mass and pellet volume with the results shown in Table 2. Based on the Harun & Afzal, 2016, a major role for pellet density, the particle size is a important factors that affected to the pellet density. Fig. 4.7a shows at the same force, the density rate decreased with increasing of particle size of EFB. The results from this study agreed well with the previous work as done by (Mo, 2014). They found that the pellets made from lower particle size exhibited higher density for all biomass. Smaller size has high

bonding formation of EFB. So it easily to be pelletized. The highest density of raw EFB pellet is is 907kgm³, while the lowest density of raw pellet is 623kgm^3 . In the past research (Hui Li *et al.,* 2012), the higher compaction pressure or higher die temperature is required to increase the density of torrified pellets. Higher density due to more pressure exhibit high solidity of pellet. The trend data analysis shows the density of torrefied EFB pellets in Fig. 4.7b decreased with increasing of degree of torrefaction temperature at the same residence time. As shown in the previous the reason why decreasing of pellet density is mainly related to the loss of chemically bonded water and low-melting point compounds which act as a binding agent when softened at 100°C. In previous research, density of torrefied EFB pellets must be lower than raw EFB pellets due to loss of chemically bonded water and low melting point compounds during torrefaction (Hui Li *et al.,* 2012). However, the result shows the highest density of torrefied EFB pellet is 974kg/m^3 , while the lowest density of torrefied EFB pellet is 574kg/m^3 . For comparison, the torrefied sample shows the low density compared with raw sample. Due to the formation of more inter-particle gaps and voids by torrefaction is the effect of why the density of torrefied pellets is about 10% lower than pellet made from raw before moisture absorption as shown in previous research (C. Wang *et al.*, 2013). Based on the previous study, R^2 was defined as coefficient of determination and the benefits of this model was easily interpretable measure for the goodness of fit of the estimated model and the fast served (Reisinger, 1997). Among of all sample from Fig. 4.7a, the $R²$ value were the perfect lines because the value near to the 1 and the best selected value from 750 μ m as a particle size of sample, which is comes out 0.976 as a \mathbb{R}^2 value. While for the Fig. 4.7b, the best selected value among all sample from 250°C as a degree temperature and 5kN as a force while conducting sample, which is come out 0.9306 as a \mathbb{R}^2 value.

Figure 4.7b : Density of torrefied EFB pellet

4.4.2 Effect of moisture adsorption of raw and torrefied EFB pellets

Moisture adsorption by dry the pellets at 30°C in oven for 2 hours was measured, with the average results shown in Fig. 4.8a and b. It is seen that the moisture adsorption capacity of EFB decreased significantly after torrefaction. Fig. 4.8a shows increasing of particle size, their effect on moisture adsorption become lower due to at larger size has high moisture content, so their adsorption is slowly. While based on increasing of force, the water adsorption rate decreased because more pressure create a high stability of pellet during pelletization. The result shows the high moisture content of raw sample at 250µm with force 1kN which is 10.38 wt% but as shown in previous research (M. Puig-Arnavat *et al.,* 2016), the high moisture content above optimum it gives negative effect on pellet density. Meanwhile, the low moisture content of raw sample at $750\mu m$ with force 5kN which is 3.57 wt%. Fig. 4.8b also shows that increasing of degree of temperature and residence time, the water adsorption rate decreased. Moisture adsorption becomes low due to the sample with the high temperature has high of drying and the sample become more brittle during torrefaction process. So, it has low quantity of water. The result shows the high moisture content of torrefied sample at 200°C with the residence time 20min and force 1kN which is 5.91 wt%, while the low moisture content at 300°C with the residence time 60min and force at 5kN which is 0.09 wt%. For comparison, the moisture adsorption of torrefied sample is lower than raw sample and as shown in previous research (Byeong-Ill Na *et al.,* 2013), the advantages of torrefaction includes higher calorific value or energy density, lower moisture content and better grinding. Among of all sample from Fig. 4.8a, the R^2 value were the perfect lines because the value near to the 1 and the best selected value from 250micron as a particle size of sample, which is comes out 0.9861 as a R^2 value. While for the Fig. 4.7b, the best selected value among all sample from 300°C as a degree temperature and 1kN as a force while conducting sample, which is come out 1.0 as a \mathbb{R}^2 value.

Figure 4.8a : Effect of weight loss on moisture adsorption raw EFB pellets

Figure 4.8b : Effect of weight loss on moisture adsorption torrified EFB pellets

4.4.3 Effect of energy consumption of raw and torrefied EFB pellets

Energy consumption associated with compression of single pellet for raw and torrefied EFB pellets are presented in Fig. 4.9a and b, respectively, as a function of force, torrefaction temperature and residence time. The trend data analysis of raw and torrefied EFB pellets in Fig. 4.9a and b shows increased trend with wide ranges of sizes and densities. Larger size becomes harder to make a pellet. Higher energy input is needed to densify the torrefied EFB pellets than raw EFB pellets, and the energy requirement increasesd with increasing the degree of torrefaction. During torrefaction, unstable hemicelluloses mainly decomposed while lignin only lost slightly. Therefore, the lignin content in the torrefied EFB was expected to be higher. If lignin is the main binding agent during pelletization, the torrefied EFB should easier to be pelletized (Hui Li *et al.,* 2012). However, our results showed the opposite trend. This may be due to the removal of most low-melting or low-softening point components during pelletization and torrefaction process. Hydrogen bonding at lignin and hemicellulose surface areas is considered as the main binding in the pelletization process of raw EFB pellets while glycerol is the main binding agent in the pelletization process for torrefied EFB pellets. Decomposition of hemicellulose and lignin reduced the plasticity of raw sample contributes to increase energy consumption during compression as shown in previous research (Hui Li *et al.,* 2012). Early study showed the torrefied EFB could not be pelletized without glycerol, compared to pelletized raw EFB. Higher die temperature is needed to make a rigid torrified pellets. Fig. 4.9a shows the highest energy consumption of raw sample at 750mic and 5kN force which is 80.68 kJ/kg, while the lowest energy consumption of raw sample at 250mic and 1kN force which is 11.24 kJ/kg. Fig. 4.9b also shows the highest energy consumption of torrefied pellet at 300°C with the residence time

60min and 5kN force which is 97.01 kJ/kg, while the lowest energy consumption of torrefied pellet at 200°C with the residence time 20min and 1kN force which is 19.01 kJ/kg For comparison, the torrefied sample consume more energy than raw sample to be pelletized the pellet. Among of all sample from Fig. 4.9a, the R^2 value were the perfect lines because the value near to the 1 and the best selected value from 750micron as a particle size of sample, which is comes out 0.9827 as a R^2 value. While for the Fig. 4.9b, the best selected value among all sample from 200°C as a degree temperature and 5kN as a force while conducting sample, which is come out 0.9979 as a \mathbb{R}^2 value.

Figure 4.9b : Effect of energy consumption torrified EFB pellets

4.4.4 Effect of Meyer hardness of raw and torrefied EFB pellets

The average Meyer hardness was measured for selected pellets, with average results shown in Fig. 4.10a and b. The hardness of pellet made at same pelleting condition decreased with the increasing of particle size of sample. The low hardness of pellet may indicate the existence of pore spaces and gaps in the pellet which reduces the pellet resistance to deformation as shown in previous research (Hui Li *et al.,* 2012). Fig. 4.10a shows that the hardness of raw sample decreased with increasing of particle sizes. The highest hardness of raw sample is at 250mic with force 5kN which is 10.77N/mm², while the lowest hardness is at the sizes 750mic with force 1kN which is 2.20N/mm². Fig. 4.10b shows that the hardness of torrefied sample decreased with the increasing of degree of torrefaction and residence time. The highest hardness of torrefied sample is at 200°C with the residence time 20min and 5kN force which is $9.71N/mm^2$, while the lowest hardness is at

300 $^{\circ}$ C with the 60min residence time and different force, 1kN which is 1.80N/mm². For comparison, the hardness of torrefied sample is lower than raw sample. Due to the amount of available hydrogen bonding sites is reduced during torrefaction. So that the strength of torrefied pellets is lowered compared to pellets made from raw as shown in previous research (C.Wang *et al.*, 2013). Among of all sample from Fig. 4.10a, the R^2 value were the perfect lines because the value near to the 1 and the best selected value from 750micron as a particle size of sample, which is comes out 0.9398 as a \mathbb{R}^2 value. While for the Fig. 4.10b, the best selected value among all sample from 250°C as a degree temperature and 1kN as a force while conducting sample, which is comes out 0.9995 as a R^2 value.

Figure 4.10a : Meyer hardness of raw EFB pellets

Figure 4.10b : Meyer hardness of torrified EFB pellets

CHAPTER 5 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The pelletization process of raw and torrefied EFB produced from Universal Testing Machine (UTM) by single pellet press (SPP) and the compression testing was used to determined the physical and mechanical properties. The pelletization process from raw and torrefied EFB was characterized to identify the physical properties like density and moisture adsorption test and also to determined the mechanical properties like energy consumption and Meyer hardness test. Energy consumption of torrified sample were significantly higher than raw sample at the same pelleting condition. The density of torrefied pellet was higher than raw pellet, while the torrefied pellet density decreased with increasing degree of torrefaction temperature and residence time. The hardness of pellet was reduced in the torrefaction process, while the hardness of pellets increased with the increasing of torrefaction but for the increasing residence time, the hardness becomes decreased. However, the moisture adsorption of torrefied pellet was lower than pellet from raw sample, while the moisture adsorption of torrefied pellets increased with increasing the severity of torrefaction. As a conclusion, based on data shows from this study, the production of pellet, physical properties and mechanical properties from raw and torrefied EFB was successful characterized.

5.2 Recommendations

 In the future, if this study is to be continued, there are several recommendations that can be considered. For example, exploration of heating rate by changing of heating rate controller. And then for the more production and quality of pellet, the another technique can be used for example pelletiser machine which is can produced more pellets within shorter time. Mold of pellets also can be fabricate into bigger size for avoiding the damage to the mold or fabricate the two mold of the same sizes for producing the pellet more faster.

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A.1 RAW DATA

A.1.1 Density

Displacement (mm) 16.889 14.722 20.041 15.722 16.388

Time (min) 60 60 60 60 60

A.1.4 Meyer hardness

A.2 TORREFIED DATA

Volume (cm³

) 0.7069 0.7069 0.7069 0.7069 0.7069 0.7069

A.2.2 Moisture Adsorption

APPENDIX B – GALLERIES

Top Left : Raw EFB were grinding to turn into powder form at wood laboratory.

Top Right : Process of conducting torrefaction process in Materials Science Laboratory

Bottom : The appearance sample of EFB after sieving process at 3 size which is 250mic, 500mic and 750mic.

C.1 Sample preparation and testing of raw pellet

C.2 Sample preparation and production of torrefied EFB

C.3 Sample preparation and testing of torrefied pellet

