PRETREATMENT OF PALM OIL WASTE VIA TORREFACTION PROCESS

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PRETREATMENT OF PALM OIL WASTE VIA TORREFACTION PROCESS

HAZRINA BT ABDUL HALIM

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering

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SUPERVISOR’S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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Dedication

I dedicate my dissertation work to my family and friends. A special feeling of gratitude to my loving parents, Abdul Halim bin Husain and Halijah binti Muda. I also dedicate this dissertation to all my friends who have supported me throughout this works.

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ABSTRACT

Recently, the demand of energy increase as the population of the world keep increasing from years to another years. However the energy sources is not sufficient to survive with the demands. Malaysia is experiencing drastic growth in population and economy and requires exploring alternative energy sources to cope its population and commercial energy demand. Biomass as the fourth largest energy resource in the world is abundant in the country. Malaysia is one of the largest producer of palm oil industries have generated huge amount of biomass from palm oil. There are many types of biomass residue generated by the palm oil industry which includes fronds, trunks, empty fruit bunches (EFB), palm mesocarp fiber (PMF), and palm kernel shell (PKS). In general, oil forms about 10% of the whole oil palm trees while the other 90% remains as biomass. All those biomass are either utilized or discarded at the plantation or palm oil mills. In this study, three types of biomass were treated by using torrefaction process. Torrefaction process is strongly depended on thermal decomposition behaviour and composition of lignocellulosic constituents. Based on the torrefaction process, the moisture contents decreases of as temperature increases. The mass yield of EFB, PKS and PMF decreases as the temperature increase. For EFB, increasing of the temperature resulted in higher heating value. However, the heating value of PMF and PKS show the highest value at temperature 270°C. Palm mesocarp fiber and palm kernel shell exhibit excellent energy yield values which are higher than 90%. Empty fruit bunch, on the other hand, exhibited a rather poor energy yield of 70%.

ABSTRAK

Kebelakangan ini, permintaan daripada peningkatan tenaga sejajar dengan penduduk dunia yang meningkat dari tahun ke tahun. Walau bagaimanapun, sumber tenaga tidak mencukupi untuk terus memenuhi permintaan. Malaysia sedang mengalami peningkatan populasi serta ekonomi yang mendadak dan memerlukan penerokaan sumber tenaga alternatif untuk menampung penduduk dan permintaan tenaga komersial. Biojisim adalahsumber tenaga yang keempat terbesar di dunia dan boleh didapati dengan kuantiti yang banyak di negara ini. Malaysia adalah salah satu daripada pengeluar terbesar dalam industri minyak sawit dan telah menjana sejumlah besar biojisim daripada pokok kelapa sawit. Terdapat banyak jenis sisa biojisim yang dihasilkan oleh industri minyak sawit termasuk, pelepah, batang, tandan buah kosong (EFB), gentian mesokarpa sawit (PMF), dan tempurung isirong sawit (PKS) .Umumnya, kira-kira 10% daripada keseluruhan pokok kelapa sawit menghasilkan minyak manakala 90% lagi akan kekal sebagai biojisim. Semua biojisim ini akan digunakan atau dibuang di kilang-kilang minyak sawit atau ladang. Dalam kajian ini, tiga jenis biojisim telah dirawat menggunakan proses *torrefaction*. Proses t*orrefaction* amat bergantung kepada tingkah laku penguraian terma dan komposisi lignoselulosa. Berdasarkan proses *torrefaction* yang dijalankan, kadar kelembapan menurun apabila suhu meningkat. Hasil jisim bagi EFB , PKS dan PMF berkurangan sejajar dengan peningkatan suhu. Manakala untuk EFB , peningkatan suhu menyebabkan peningkatan yang lebih tinggi nilai pemanasan. Walau bagaimanapun , nilai pemanasan PMF dan PKS menunjukkan nilai yang paling tinggi pada suhu 270 °C. Gentian mesokarpa sawit dan tempurung isirong sawit mempamerkan nilai hasil tenaga yang sangat baik iaitu lebih tinggi daripada 90%. Namun, buah tandan kosong hanya menghasilkan jumlah tenaga yang agak rendah iaitu sebanyak 70 %.

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LIST OF ABBREVIATIONS

AWER	Association of Water and Energy Research Malaysia
CBBR	Center for Biofuel and Biochemical Research
EFB	Empty fruit bunch
FFB	Fresh fruit bunch
LCSB	LKPP Corporation Sdn Bhd
LKPP	Lembaga Kemajuan Perusahaan Pertanian
MTOE	Metric of oil equivalent
PKC	Palm kernel cake
PKS	Palm kernel shell
PMF	Palm mesocarp fiber
UNITEN	Universiti Tenaga Nasional
UTP	Universiti Teknologi PETRONAS

1 INTRODUCTION

1.1 *Motivation and problem statement*

Energy is one of the most vital elements that required in our daily life which is used for transportation, telecommunication and industrial activities that influence the economic growth. Today, energy crisis turn out to be a serious threat towards sustainability for developing countries since their energy demand is growing more rapidly than developed countries. In Malaysia, the electricity energy sector is forecasted growth, the demand is expected to escalate from 91,539GWh in year 2007 to 108,732GWh in year 2011 (VGR, et al., 2010). By year 2020, the energy demand in Malaysia is projected to reach 116 MTOE based on an annual growth rate of 8.1% (Shafie, et al., 2011). The Malaysian energy sector is still heavily dependent on non-renewable fuel such as fossil fuels and natural gas as a source of energy. However, those conventional fuels will not able to sustain for another 100 years and limited (Mekhilef, et al., 2011). Those non-renewable fuels are finite and gradually depleting and also contribute to the emission of greenhouse gases (Jamaludin, 2010). Thus, we need to find another alternative in substituting the non-renewable energy to the renewable and other alternative energy. Therefore, biomass energy is the preeminent substitute to petroleum-derived energy and the most suitable as a backup for sustainable energy development. By the year 2050, biomass is expected to become the most prominent renewable energy source with a four-fold increase to 23% of the total world primary energy (Umar, et al., 2014). Malaysia, is a tropical country that experiences hot and wet weather throughout the year. Those climate has encourages the growth of the oil palm and presently Malaysia was occupied with million hectares of land with palm oil plantation generating huge quantities of biomass (Pei, et al., 2012). It clearly shows that biomass from palm oil industries will be a very promising alternative as a source of raw materials (Shuit, et al., 2009). The abundance availability of palm oil waste in country since Malaysia is one of the largest producers of palm oil industry will lead to an average of 53 million tonnes of residues each year and it is projected to rise to 100 million dry tonnes of palm oil biomass by the year 2020 (Umar, et al., 2014).

Oil palm, or also known as *Elaeis guineensis* is a perennial tree crop (Shuit, et al., 2009)(Yusoff, 2004), which is cultivated extensively in the humid tropical land. There are many types of biomass residues generated by the palm oil industry which includes fronds, trunks, empty fruit bunches (EFB), palm mesocarp fiber (PMF), and palm kernel shell (PKS) (Wei & Po, 2010). In general, oil forms about 10% of the whole oil palm trees while the other 90% remains as biomass. All those biomass are either utilized or discarded at the plantation or palm oil mills. Biomass has the highest potential to contribute to energy needs and replace the existing conventional fuel. Since Malaysia is the largest producer of palm oil industry, so the main raw materials used in this study were comes from palm oil industry. Besides of the abundance availability of sources, it also eco-friendly and can reduce the effect of global warming. Burning of palm oil biomass instead of fossil fuels as an energy source, it will displace a certain amount of carbon that would have been released to the environment (Shuit, et al., 2009). Reduction in carbon emission to the environment is crucial to prevent further global warming. However, biomass has its own limitation properties that need to overcome in order to utilize biomass efficiently. To develop energy from biomass, various conversion technologies such as physical, chemical, thermal, and biological methods have been utilized. The most commonly technique used is thermal conversion like direct combustion, pyrolysis, gasification and liquefaction (Zhang, et al., 2010). But, many problems occur in order to utilize raw biomass because of its characteristics. From the previous studies, torrefaction process is the most suitable pretreatment process that compatible with the biomass characteristic itself. Energy density of pretreated biomass could be increased by 8-36% depending on torrefaction conditions (Uemura, et al., 2011). Torrefaction is a promising technique to improve the performance of biomass for energy utilization. As can be seen, most studies only focusing on several types of biomass and parameter such as energy yield or mass yield based on difference temperature. Hence, more research is required to study more about torrefaction process using different biomass and different parameters.

1.2 Objectives

The objectives of this work are:

- To study about pretreatment of palm oil waste via torrefaction process.

- To study the influence of torrefaction process on physical and chemical properties of biomass.

1.3 Scope of this research

The following are the scope of this research:

i) Construction of experimental rig for palm oil waste; empty fruit bunch, palm mesocarp fiber and palm kernel shell performance analysis

ii) Experimental analysis of torrefied biomass based on mass and energy yield, moisture content using temperature dependence parameter.

2 LITERATURE REVIEW

2.1 Overview

Torrefaction, a thermochemical conversion process, has gained global attention as a viable pretreatment technology for biomass feedstock in gasification and co-combustion processes (Olumuyiwa, et al., 2014). Torrefaction process is a thermal pre-treatment process at temperature ranged of 200 ºC to 300 ºC (Aziz, et al., 2012) (Chen & Kuo, 2011) (Prins, et al., 2006) and resulting of numerous improvement on torrefied biomass such as reduce the moisture content, increase the energy density, and increase the heating value (Uemura, et al., 2012) . Therefore, the purpose of this project is to study about the influence of torrefaction process on physical and chemical properties of biomass. Biomass, is biological material derived from living organisms such as wood and herbaceous material (Tumuluru, et al., 2011). It is used as renewable energy source to generate electricity or to produce heat. However, several weakness of biomass have limited its wider application in energy generation. These include low heating value, high moisture content, low energy density and low grindability properties (Jaafar & Ahmad, 2011). It, therefore often needs to pre-treat to improve the weakness of the biomass. Torrefaction promises to deliver a solid biofuel which has superior characteristics such as increase the heating value and energy density, lower the moisture content and also in terms of handling, milling and transport. This has potential to vastly improve the competitiveness of biomass as renewable energy carrier (Batidzirai , et al., 2013).

2.2 Introduction

Energy is a property of objects that cannot be create or destroy but can be transferred among them via fundamental interaction. It is one of the furthermost elements that essential in our daily life. Energy is crucial to all aspects of development from powering manufacturing and modernization of agricultural sectors to providing electricity to run schools and health facilities, yet the impact of its production, distribution and use grows more severe with every decade (Mohamed & Lee, 2004). Most of daily routine are consume energy such as transportation, telecommunication as well as industrial activities and those activities influence the growth of economy. In Malaysia, more than 50% of domestic consumers use less than 200 kilowatt-hour (kWh) of electricity monthly. However, the percentage of domestic consumers that use less than 200 kWh electricity monthly steadily decrease since the electric product become cheaper. Increase the electricity consumption means increasing the living cost and at the same time will impact the environment.

Nowadays, energy crisis turn out to be a serious threat towards sustainability for developing countries since their energy demand is growing extra speedily than developed countries. In Malaysia, electric sector is depending on fossil fuels. Regrettably, there are a number of serious problems with burning these fuels to provide energy. It will cause significant damage to the natural and built environments as well as human health. Burning fossil fuels releases a number of chemical into air such as carbon dioxide, nitrogen dioxide, heavy metals, sulfur dioxide and volatile organic compounds. Those chemicals released through burning of fossil fuels can lead to acid rain, which can cause damage to plants and buildings. Besides that, changing the proportion of carbon dioxide in the atmosphere can lead to changes in the climate which could affect weather patterns and sea level too. Furthermore, burning of fossil fuels will cause health problem. Heavy metals that are released into air as a result from combustion of fuels can lead to higher rates of cancer and increased risk of respiratory illnesses in the surrounding area. And, the most serious problem of fossil fuels is that they are non-renewable (RP, 2007). Thus, a drastic step should be taken in order to overcome the problem of using fossil fuels as conventional sources. Therefore, many research have been carried out to find an alternative to substitute the conventional source. As a result, biomass energy is the preeminent substitute to petroleum-derived energy and the most suitable as a backup for sustainable energy development.

2.3 Biomass and its characteristics

Biomass is an organic material that derived from botanical or biological. It is a non-fossilized fuel source that is biodegradable. It also sustainable and renewable energy sources and has highest potential to contribute to energy needs and replace the existing conventional fuel since it is abundant, clean and carbon dioxide neutral. It is the third largest primary energy sources after coal and oil (Zhang, et al., 2010). Biomass can be divided into two main class which are virgin biomass and waste biomass. Virgin biomass comes from terrestrial or aquatic source. Meanwhile, for waste biomass comes from industrial waste, municipal waste, agricultural solid waste and forestry waste. Table 2-1 shows the biomass classification and their example.

Table 2-1: Biomass Classification (Tumuluru, et al., 2011)

Virgin Biomass	**Waste Biomass**
• Terrestrial • Aquatic	• Municipal waste • Agricultural solid waste • Forestry residue • Industrial waste

Biomass is made up of cellulose, hemicellulose, lignin, extractives and inorganics (ash) with different physical and chemical properties due to its diverse origin and species. Cellulose with a molecular weight of about 100,000 is a polymer with linear chains of glucopyranose units linked to each other by its 1,4 carbon atoms in the β-configuration. Hemicellulose, the second major constituent with lower molecular weight, is a mixture of various polymerized monosaccharides such as glucose, mannose, galactose, xylose, arabinose, methyl glucuronic and galacturonic acid residue. A highly branched polymer attached with polysaccharides, lignin, is composed of phenyl propane based monomeric units linked together by several types of ether linkages and also various kinds of carbon-carbon bonds (Kong, et al., 2014). Not only that, biomass also have their own characteristics that cause the several problem in order to generate energy from it. Consequently, the biomass need to pre-treat before further process to generate energy is taken. There are many problem regarding the biomass such as higher energy consumption during collection, low energy density, low calorific value, and high moisture content. Various conversion technologies have been explore in order to utilize the raw biomass caused by its characteristics. The most common technique is thermal conversion, the technique that based on the temperature as the parameter. Example of thermal conversion applied are direct combustion, pyrolysis, gasification and liquefaction (Zhang, et al., 2010). However, a pre-treatment method called torrefaction is found to be effective process to overcome the limitation properties of raw biomass.

Torrefaction is a promising pretreatment technology with potential to make a major contribution to the commodification of biomass. Torrefaction is a process of thermal pretreatment to pretreat biomass that maintained by an operating temperature ranging from 200ºC to 300ºC (Jaafar & Ahmad, 2011). It is carried out under atmospheric pressure, in the absence of oxygen and in the flow of nitrogen gas. This process strongly depended on the decomposition temperature of the lignocellulosic constituents in biomass. There are many advantages of torrefaction to the raw biomass. Table 2-2 show the biomass characteristics and improvements through torrefaction pretreatment.

Table 2-2: Biomass characteristic and improvement through torrefaction pretreatment (Batidzirai , et al., 2013)

Biomass Characteristic	***Improvement through torrefaction pretreatment***
Low heating value (due to low fixed carbon content of about 45% and relatively high moisture content, typically about 50%)	Increase fixed carbon; the fixed carbon content of torrefied biomass is high (25-40% depending on the reactions conditions). Combustion properties: torrefied biomass burns longer due to larger percentage of fixed carbon
Lower energy density than fossil fuels (2-4 GJ/m^3)	Densification: Torrefied and pelletised biomass (TOPs) has high energy density of 18-20 GJ/m^3. It contains 40-80% of the original mass while retaining 80-96% of the original energy of the dry biomass.
Hydrophilic and hygroscobic (absorbs moisture during storage and transportation)	Hydrophobicity: torrefied biomass becomes hydrophobic. The equilibrium moisture content of torrefied biomass is very low (from 1 to 3%), but depends on severity of torrefaction
Heterogeneous characteristics (wide range of shapes, sizes and types) and quality	Torrefaction produces a solid uniform product, as volatiles and moisture are eliminated. On grinding the particle size distribution, sphericity and particle surface areas become similar to coal. It also results in improved chemical composition making it more suitable for fuel applications.
Low combustion efficiency	Reduced oxygen: torrefaction reduces the O/C ratio and this makes a biomass better suited for gasification. Torrefied biomass also produces less smoke during combustion since smoke causing

	volatiles are driven off during torrefaction.
Tough and fibrous, difficult to pulverize	Improve grindability: torrefied biomass requires less energy to grind compared to raw biomass.
Biodegradable: due to moisture and environmental stress	Stable: torrefied biomass has very low moisture and can therefore be transported and stored for long periods without any biological degradation.
Small quantity scattered over many sources locations and seasonal availability	Biomass can be torrefied and pelletised in decentralised locations and stored for long period without an impact of its quality. It also improve the economics of transportation and handling biomass.

2.4 Palm Oil Waste as Biomass

Palm tree usually have a vertical trunk and feathery leaves known as palm frond. Every year around 20-40 new leaves are grown. Bunches of palm fruit develop between trunk and base of the new fronds. After 5-6 years of plantation, the first crop of fresh fruit can be harvested and each tree can provide palm fruit for 25-30 years. Each fruit has a spherical shape and black color before turning to orange-red when ripe (Hosseine & Wahid, 2014). Normally, about 10% of the whole palm oil trees will produces oil while the other 90% remains as biomass. Fresh fruit bunch contains only 21% palm oil, while the rest are 14-15% fiber, 6-7% palm kernel, 6-7% shell and 23% empty fruit bunch which left as biomass (Aziz, et al., 2012; Fauzianto, 2013). Figure 2-1 illustrates the typical palm tree and fresh fruit bunch (FFB).

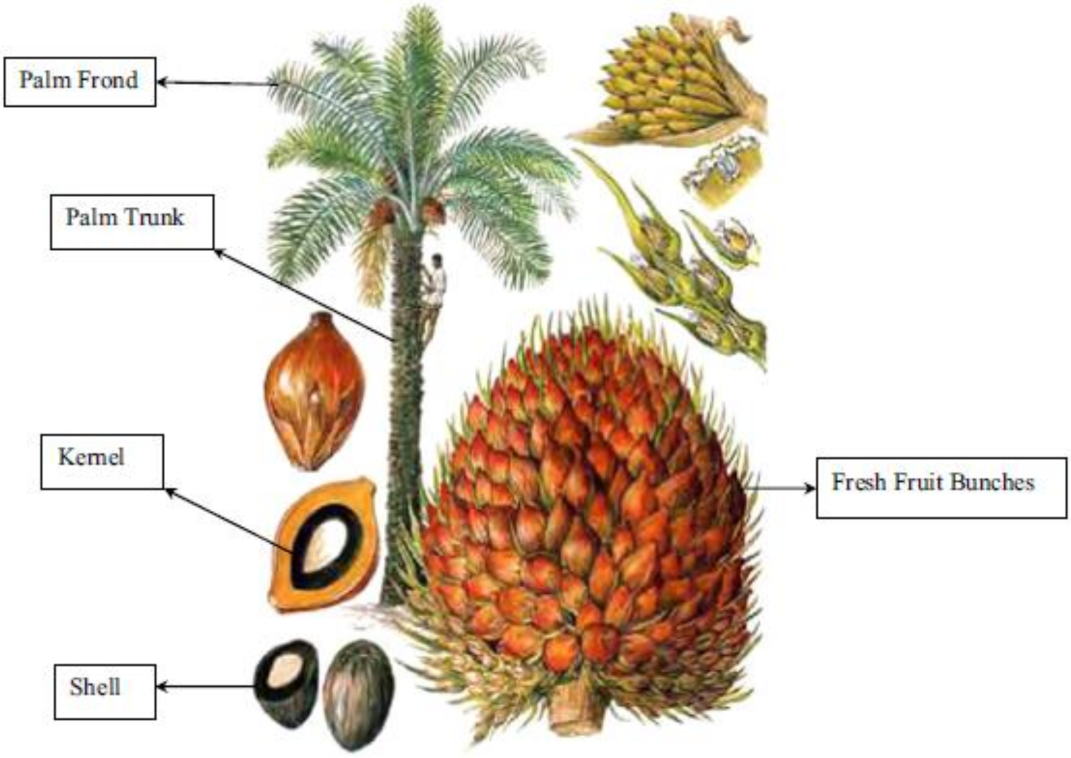


Figure 2-1: Typical palm tree and FFB (Hosseine & Wahid, 2014)

In recent decades, Malaysian government has introduced biomass as the fifth fuel resource after petroleum, gas, coal and hydro (Yaakob, et al., 2011). The most important biomass resources in Malaysia are agricultural waste, effluent sludge, domestic waste as well as wood chips. However, due to specific weather circumstances, palm oil biomass has been developed in huge quantities in Malaysia. Since the abundance of palm oil biomass, it seem to have a great potential to be utilized in different industries. Some solid residues and leftovers like EFB, PMF and PKS from palm oil fruit remains in the harvesting process and also palm oil mills. Therefore, the palm tree plantation in Malaysia are continuously increasing due to Malaysia government strategies for palm-oil based biodiesel production. Figure 2-2 demonstrates the types of different by-products generated annually from palm oil industry as palm biomass in Malaysia.

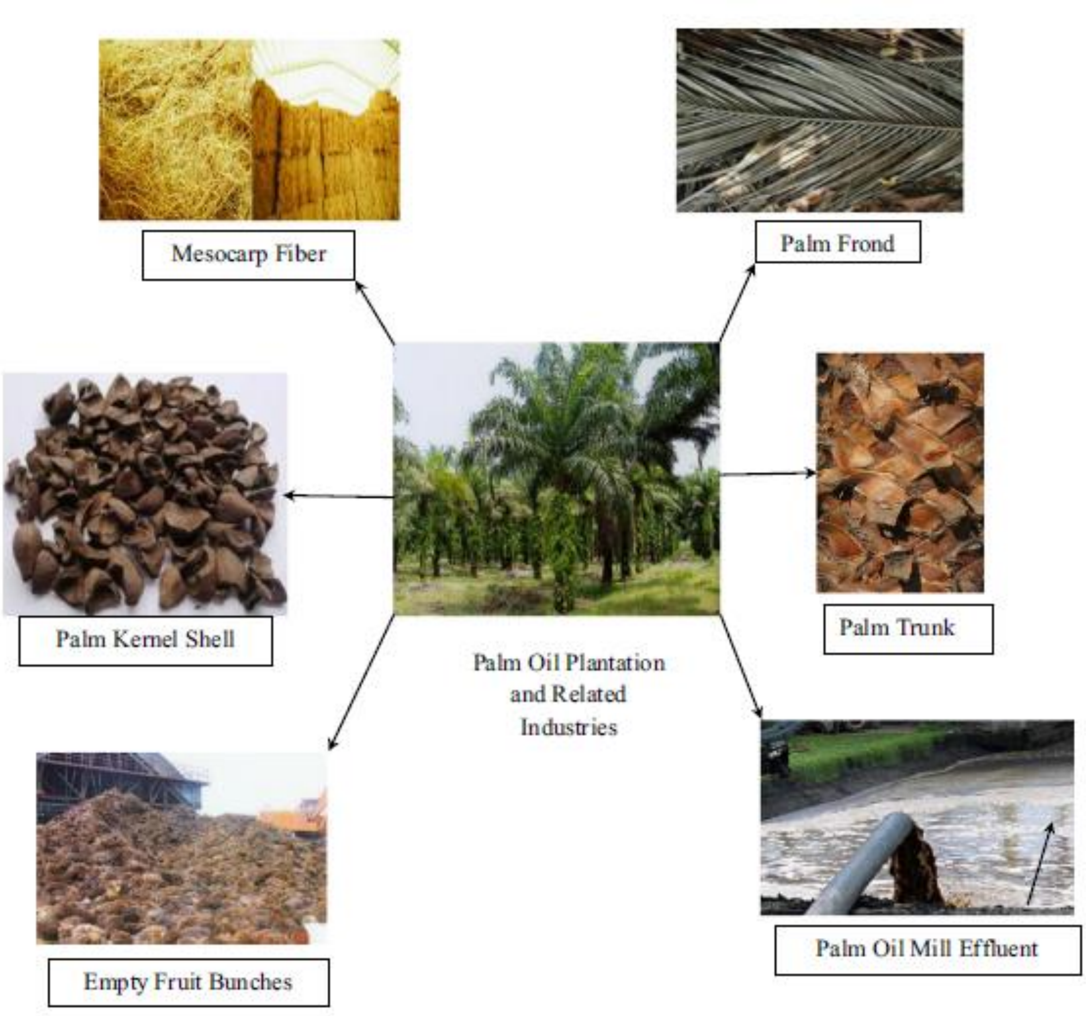


Figure 2-2: Summary of biomass by-product generation in palm oil industries in Malaysia.

As illustrated in Figure 2-2, a lot of biomass by-product has been generated in palm oil industries in Malaysia. Therefore, abundant waste biomasses are turned into renewable energy or value added product. Table 2-3 shows the types of renewable energy in Malaysia and its energy value. From the table shown, palm oil biomass is the second largest that contribute in energy after forestry residues.

Table 2-3: Type of renewable energy source in Malaysia and energy value (Sumathi, et al., 2008)

Renewable energy source	**Energy value in RM million**

	(annual)
Forest residues	11,984
Oil palm biomass	6379
Solar thermal	3023
Mill residues	836
Hydro	506
Solar	378
Municipal waste	190
Rice husk	77
Landfill gas	4

The production of palm biomass was approximately 87 Mt in 2010, although this value excludes oil palm fronds and trunks, which would further increase the amount of biomass produced by the palm oil industry. The potential energy that can be generated by palm oil biomass is shown in Table 2-4. Those amount of energy may be wasted due to inefficient utilisation of the palm oil biomass. Recently, the government of Malaysia has set target to increase its biomass power generation capacity to 800MW by 2020, and 500MW is to be generated from palm oil biomass.

Table 2-4: Amount of biomass available as 2010 (Qin Ng, et al., 2012)

Biomass available from palm oil industry	**Quantity (Mt/y)**	**Potential energy (MTOE/y)[a]**
Empty Fruit Bunch	21.27	9.55
Mesocarp Fiber	10.80	4.92
Palm Kernel Shell	4.98	2.39

Palm Oil Mill Effluent	49.85	20.23
Total	86.90	37.09

[a] 1 Mt of oil equivalent (MTOE) = 41868 MJ

2.5 Type of Palm Oil Biomass

Many type of biomass that generated from palm oil industries such as empty fruit bunch (EFB) , palm kernel shell (PKS), palm kernel cake (PKC), palm mesocarp fiber (PMF) , fronds and trunks. Yet, this study only focus on three types of biomass which are EFB, PKS and PMF.

2.5.1 Empty Fruit Bunch (EFB)

EFB is formed from fresh palm oil fruit bunch (FFB) that went through steam heating and put under threshing process which separate palm fruit from its bunch. Figure 2-3 illustrated the real picture of EFB taken from the palm oil mill.

Figure 2-3: Empty Fruit Bunch

Each biomass contains its own characteristics and application. Table 2-5 how the physical properties of EFB such as moisture content, calorific value and elementary and ash analysis. In addition, the chemical of composition of EFB has been listed on Table 2-6.

Table 2-5: Physical properties of EFB (Uemura, et al., 2011)

Moisture content	**Calorific value (MJ/kg)**		**Elementary and ash analysis**						**Form**
	Wet (HHV)	Dry (HHV)	C	H	N	S	O	Ash	
65	-	19.1	48.8	6.30	0.20	0.20	36.7	7.3	Fiber of 1 mm in diameter

Table 2-6: Chemical composition of EFB (Aziz, et al., 2012)

Component	**Hemicellulose**	**Cellulose**	**Lignin**
EFB (wt %)	35.3	38.3	22.1

As mentioned before, each type of biomass have their own physical properties. Different properties will contribute to different type of application. For EFB, it has own application. There are several application of EFB in various industries (Sumathi, et al., 2008):

1. Convert into paper-making pulp. The paper can be used as cigarette paper or bond paper for writing.
2. Used as soil conditioner in estate and plantation. It will incinerated to obtain oil palm ash (OPA) that can be used as a source of fertilizers due to its high potassium.
3. Manufacture medium density fiber board (MDF) and blackboard.
4. Used to produce glucose and xylose
5. Used as a feedstock for second generation ethanol to produce bio-oil and bio-ethanol.

2.5.2 Palm Mesocarp Fiber

An elongated cellulose that generated at the nut/fiber separator is known as palm mesocarp fiber. PMF is one of the biomass that used in this study. The physical properties and chemical composition of the PMF is shown in Table 2-7 and 2-8, respectively. Figure 2-4 show the image of PMF that generate from palm oil industries.


Figure 2-4: Palm mesocarp fiber

Table 2-7: Physical properties of PMF (Uemura, et al., 2011)

Moisture content	**Calorific value (MJ/kg)**		**Elementary and ash analysis**						**Form**
	Wet (HHV)	Dry (HHV)	C	H	N	S	O	Ash	
42	-	18.8	47.2	6.00	0.30	0.20	36.7	8.4	Fiber of 1 mm in diameter

Table 2-8: Chemical compositions of PMF (Aziz, et al., 2012)

Component	**Hemicellulose**	**Cellulose**	**Lignin**
PMF (wt %)	31.8	34.5	25.7

PMF also have its own application that can be applied in many types of industries such as agriculture as soil conditioner. There are a lots of application of PMF like (Sumathi, et al., 2008):

1. Used as a boiler fuels to generate steam for mill consumption.
2. Used as soil conditioner in estate and plantation. It will incinerated to obtain oil palm ash (OPA) that can be used as a source of fertilizers due to its high potassium.

3. Manufacture medium density fiber board (MDF) and blackboard.
4. Can be converted into value added products such as oil palm activated carbon. This palm oil activated carbon has been used to treat air toxics such as carbon monoxide (CO) and SO_x

2.5.3 Palm Kernel Shell

PKS is a crushed nuts of palm oil fruits. It is a by-product of palm oil production. It is carbonaceous solids that contain high volume percentage of carbon element and can be converted as a heat energy source. PKS also known as “Virgin Biomass”, free of chemical treatment, metallic plastics and not obvious degraded, not sticky and manageable in normal loading/discharging (Nordic , 2011). Figure 6 shown the picture of palm kernel shell.

Figure 2-5: Palm Kernel Shell

Table 2-9 shows the physical properties of PKS and the chemical composition of PKS is shown in Table 2-10.

Table 2-9: Physical Properties of PKS (Jaafar & Ahmad, 2011) (Uemura, et al., 2011)

Moisture content	Calorific value (MJ/kg)		Elementary and ash analysis						Form
	Wet (HHV)	Dry (HHV)	C	H	N	S	O	Ash	
2.88	-	18.81	46.5	5.85	0.89	0.20	42.3	4.3	

Table 2-10: Chemical composition of PKS (Aziz, et al., 2012)

Component	Hemicellulose	Cellulose	Lignin
PKS (wt %)	22.7	20.8	50.7

PKS has less application compare to PMF and EFB because of its own characteristics and properties. However, PKS can be used as a boiler fuels to generate steam for mill consumption and can be converted into value added products such oil palm activated carbon. This palm oil activated carbon has been used to treat air toxics such as carbon monoxide (CO) and SO_x **(Sumathi, et al., 2008)**.

2.6 *Torrefaction process*

Torrefaction is a process used to produce high-grade solid biofuels from various streams of streams of woody biomass or agro residues. The end product is stable, homogenous, high quality biofuel with far greater energy density and calorific value than the original feedstock, providing significant benefits in logistics, handling and storage, as well as opening up a wide range of potential uses.

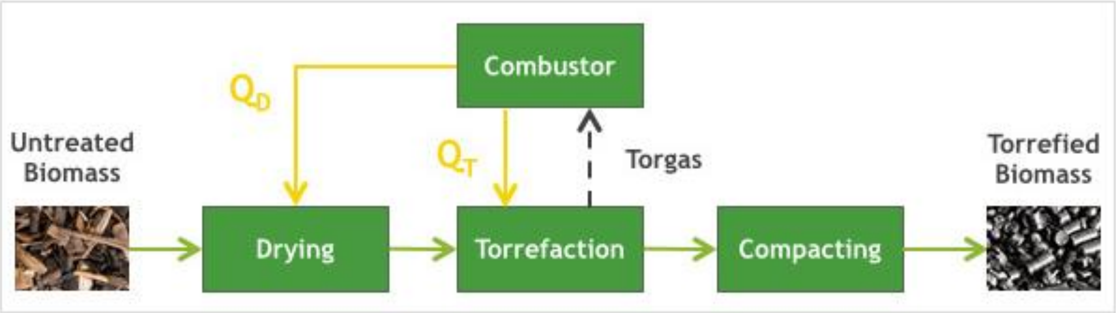


Figure 2-6: Basic Torrefaction Principle

Torrefaction is a thermal process that involves heating the biomass to the temperatures between 200 to 300 ºC in an inert atmosphere. When biomass is heated at such temperatures, the moisture evaporated and various low-calorific components contained in the biomass are driven out. During this process the hemicellulose in the biomass decomposes which transforms the biomass from a fibrous material into a product with excellent fuel characteristics.

As a result of torrefaction of biomass, high grade biofuel is produced which can be used as a replacement of coal or fossil fuels in electricity and heat production. It also can be used as fuel for gasification process in the production of high value bio-based fuels and chemicals.

2.7 Torrefaction in Malaysia

In recent decades, Malaysian government has introduced biomass as the fifth fuel resource after petroleum, gas, coal and hydro. However, before biomass can be used as the alternative resources, it must be treated because of its characteristics. There are several research that have been carried out in Malaysia about torrefaction process. One of them is torrefaction in the presence of oxygen and carbon dioxide (Saadon, et al., 2014). The research was carried out by Center for Biofuel and Biochemical Research (CBBR), Universiti Teknologi Petronas (UTP), is to study the behaviour of torrefaction diverged when the process was carried out in the presence of oxygen and carbon dioxide. The chosen biomass is oil palm kernel shell due to its abundance in Malaysia.

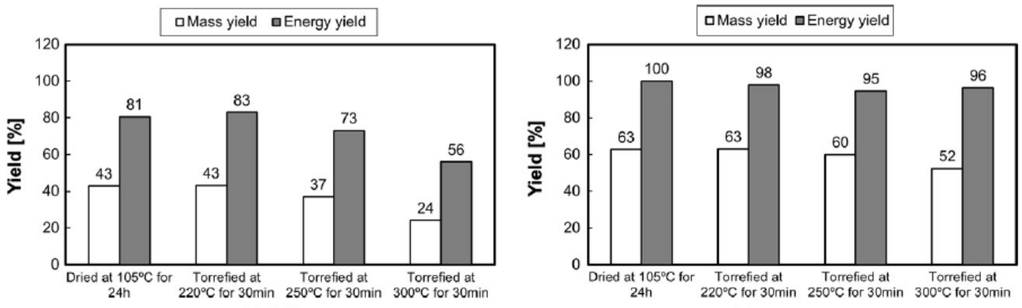
Other than that, another researcher from Universiti Tenaga Nasional (UNITEN), has developed a bench scale bbiomass torrefier to produce samples of torrefied biomass to be used for further fuel characterization such as calorific value and ultimate analysis (Mohd Jaafar, et al., 2013). Previous work on torrefaction on Malaysian biomass had been done using thermogravimetric analyser (TGA) or tube furnace apparatus as the torrefier. Those apparatus can only torrefy small amount of biomass, a maximum of 5g, at a time. So, only a small amount will produce and insufficient sample size for further characterization studies. Hence, by developing a bench scale torrefier, it could handle a larger biomass sample for characterization studies.

2.8 *Torrefaction of Palm Oil Waste*

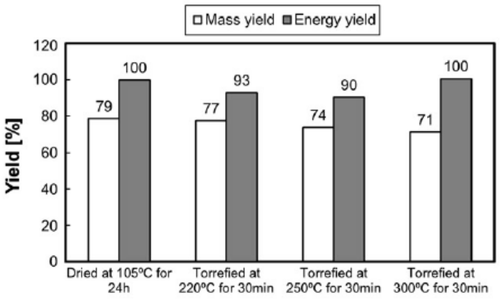
Torrefaction process has been used for high energy density on lignocellulosic biomass like EFB, PMF and PKS. It is a pretreatment process where raw material is heated in an inert or nitrogen atmosphere, at a temperature 200-300˚C. A hydrophobic condition for biomass was provided due to the removal of hydroxyl group during thermal treatment (Na, et al., 2013). Torrefaction of biomass accompanies weight loss, depending on the torrefaction condition. A high calorific value of the torrefied biomass is indicative of such weight loss during torrefaction. Therefore, torrefied biomass must be evaluated in terms of energy yield. During the torrefaction of biomass, most volatile compounds are removed from the biomass as vapour and result in a higher energy density.

After the torrefaction process, the color of EFB and PMF becomes darker as the temperature of torrefaction increase. Uemura et al (2011) reported the mass of torrefied sample treated at 220ºC, the lowest temperature showed the almost the same decrease in mass as that of dried at 105ºC. As the torrefaction temperature increases, the mass yield decrease steadily, whereas the decreasing ratio is depending on the waste type. Based on the research, EFB has the highest decreasing ratio and PKS has the lowest.

There are two main causes of the decrease in mass of the dried or torrefied samples. First is moisture loss, and another is thermal decomposition to form volatile or gaseous products such as H_2O, CO, CO_2, acetic acid and other organics (Prins, et al., 2006). Figure 4 show the distribution of mass and energy yield of three type of biomass. The temperature range is from 220ºC to 300ºC. Based on the figure, it clearly show that the energy yield for EFB more differences between the temperature ranges compare to PMF and PKS. The same goes for mass yield, where the EFB show the most differences compare to the others.



(a) EFB (b) Mesocarp Fiber



(c) Kernel Shell

Figure 2-7: Mass and energy yield for (a) EFB, (b) PMF and (c) PKS (Uemura, et al., 2011)

3 MATERIALS AND METHODS

3.1 Overview

The purpose of the research is to investigate the influence of torrefaction process on physical and chemical properties of biomass. In the present chapter, the details of materials involved and related experimental setup are discussed in detail. This chapter is divided into four sections. Section 3.2, 3.3 and 3.4 deals with the materials used for the present studies and the preparation of sample, respectively. The details on the experimental setup for torrefaction process are discussed in section 3.5. Figure 3-1 shows the overall process of the experiment.

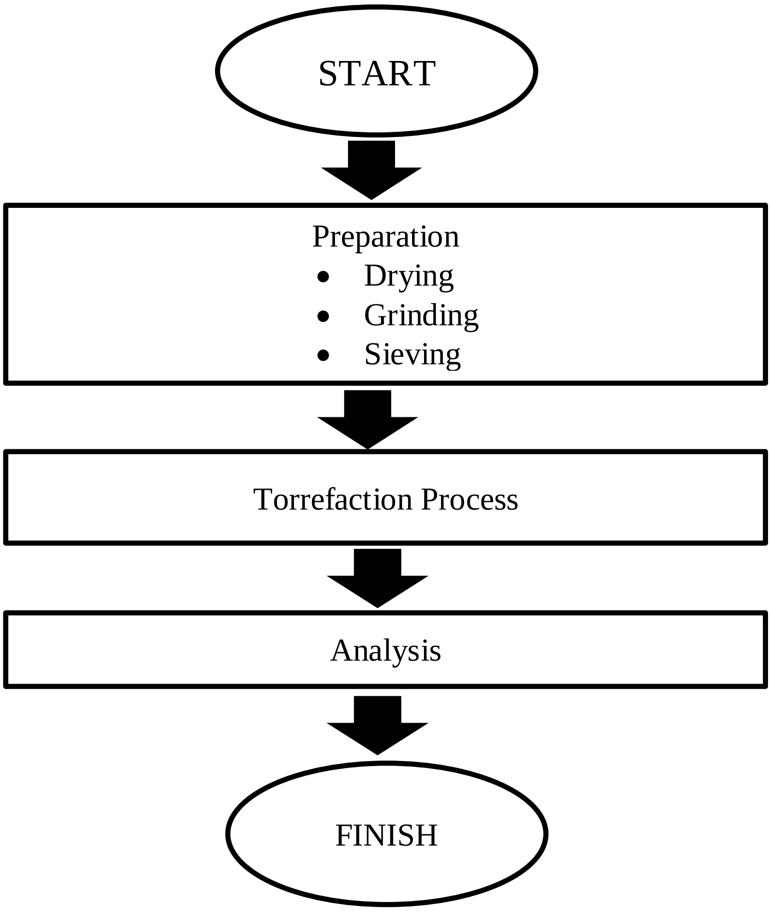


Figure 3-1: Overall process of the experiment

3.2 Materials

There are three types of biomass involves in this study which are Empty Fruit Bunch (EFB), Palm Mesocarp Fiber (PMF) and Palm Kernel Shell (PKS). About 3kg of each types of biomass were collected at Palm Oil Plantation LCSB Lepar Hilir, Gambang.

3.3 Preparation of samples

Raw biomass that collected from Lepar Hilir were prepared before it can be used in torrefaction experiment. Figure 3-2 shows the samples of raw biomass before drying process. The samples were dried in an oven under temperature 105ºC for certain time until the moisture content in the sample is less than 10%. Figure 8 shows the raw biomass before drying process.

Figure 3-2: Raw biomass before drying process

Next, the sample were grounded in order to reduce the size using grinding machine and blender. Figure 3-3 show the grinding machine that used for grinding the raw biomass. After the grinding process, the raw biomass were sieved to get the desired size about 2 to 4 mm for the experimental purpose using sieve shaker. Figure 3-4 shows the sieve shaker that used for sieving the raw biomass. Each tray of the sieve shaker has different size. For this study, only four tray was used which sized 4mm, 2mm, 1mm and also bottom tray.

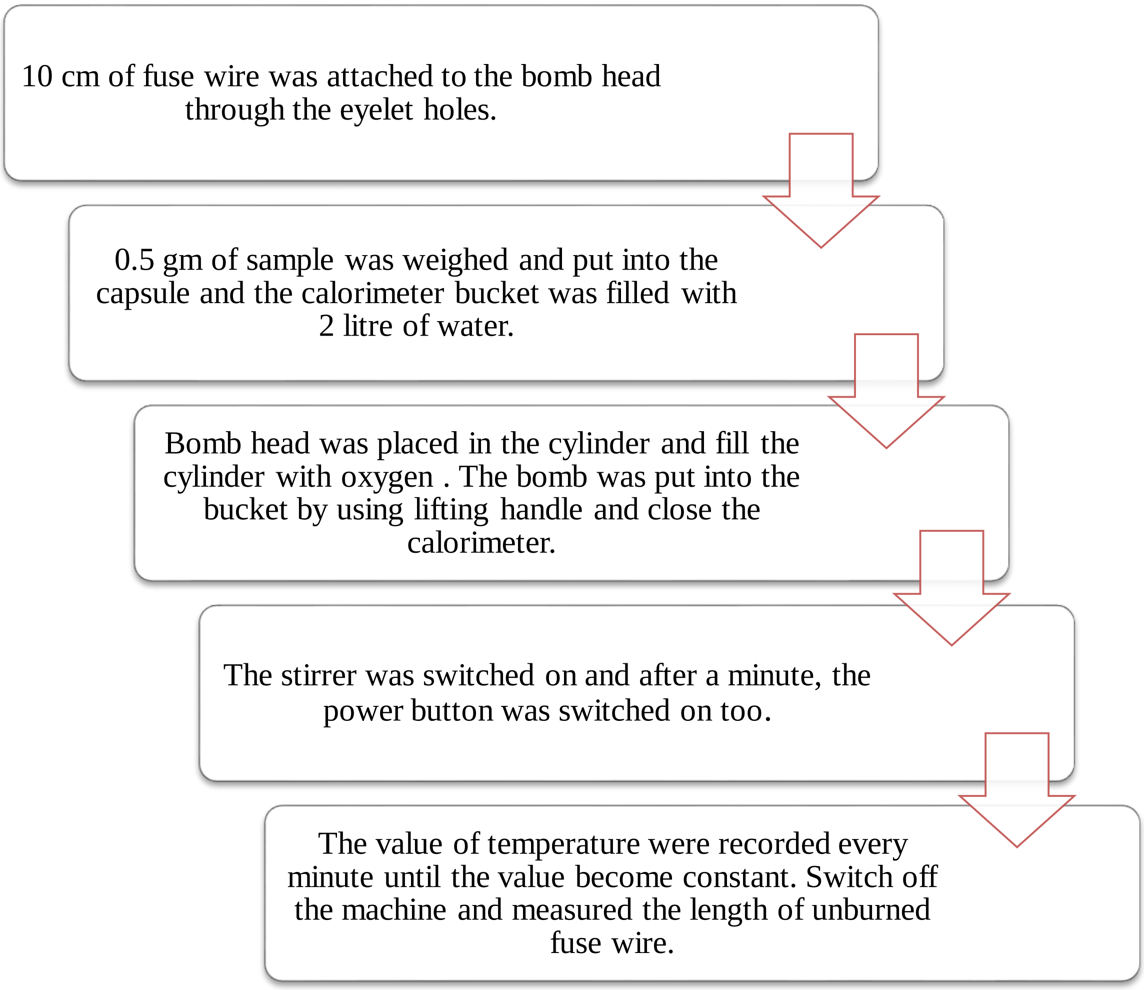
Figure 3-3: Grinding machine

Figure 3-4: Sieve Shaker

Then, after desired size of raw biomass was obtained, the heating value of raw biomass was determined using oxygen bomb calorimeter. The heating value can be determined as the energy released as heat when a compound undergoes complete combustion with oxygen. Figure 3-5 shows the bomb calorimeter that used for determine the heating value of raw and torrefied biomass.

Figure 3-5: Bomb Calorimeter

The bomb calorimeter consists of heavy-walled stainless steel reaction vessel containing weighed sample of the compound to be burned with sufficient oxygen gas. Below, Figure 3-6, listed the procedures of using bomb calorimeter.



10 cm of fuse wire was attached to the bomb head through the eyelet holes.

0.5 gm of sample was weighed and put into the capsule and the calorimeter bucket was filled with 2 litre of water.

Bomb head was placed in the cylinder and fill the cylinder with oxygen . The bomb was put into the bucket by using lifting handle and close the calorimeter.

The stirrer was switched on and after a minute, the power button was switched on too.

The value of temperature were recorded every minute until the value become constant. Switch off the machine and measured the length of unburned fuse wire.

Figure 3-6: Methodology of bomb calorimeter

3.4 Chemicals

Only nitrogen gas will involve in the whole study of torrefaction process. Nitrogen gas is required to ensure an inert atmospheric condition by eliminating the oxygen from torrefaction reactor.

3.5 Torrefaction using Gas Catalytic Reactor

The torrefaction experiment was carried out in a stainless steel vertical tubular reactor with an internal diameter of 22.4 mm and a length of 500 mm. A prescribed amount of biomass (around 2gm) was weighed and put on the glass wool. The glass wool was placed at the center of the reactor. After flushing the reactor with nitrogen, the temperature was raised to different desired levels of 230, 270 or 300ºC at a constant rate of 10ºC/min by a tube furnace surrounding the reactor. After 60 minutes torrefaction, the heater was turned off ant the reactor was left to cool down to the ambient temperature. Then, the torrefied sample was recovered, weighed, and determine the heating value of torrefied biomass. The torrefied sample was kept in air tight container. 0.5L/min of nitrogen was flowed through the reactor throughout the procedure. The experiment was repeated 2 times for reproducibility. Figure 3-7 shows the tube furnace with the stainless steel vertical tubular reactor.

Figure 3-7: Gas Catalytic Reactor with Vertical Tube reactor

4 RESULTS

4.1 Overview

This chapter presents a comprehensive study on the effect physical and chemical properties of biomass by using torrefaction process. Three types of biomass which are EFB, PMF and PKS was used as the main sample to observe the trend on moisture content, mass and energy yield based on various temperature. The torrefaction process was experimentally evaluated using the gas catalytic reactor, nitrogen (NO_2) gas tank as a source of NO_2 that used in the experiment to create an inert atmosphere.

In this chapter, the results that will be discuss and analyse are the result from previous research (Uemura *et. al., 2011*) who were doing a similar work as this study and within the range of quite similar parameter except the analysis of elementary and ash. Therefore, the results presented are almost similar from the previous research.

4.2 Appearance of torrefied samples

In this study, three types of palm oil waste were torrefied. In Figure 4-1, photos of raw biomass and torrefied biomass are exhibited. The colour of biomass becomes darker as the torrefaction temperature increases.

Figure 4-1: Colour differences between raw and torrefied biomass

4.3 Moisture content analysis

Moisture content or water content is the quantity of water contained in a material such as soil, rock, fruit or wood. Before carried out the torrefaction process, the moisture content of all the samples should be less than 10%. So to ensure the sample was dried, the moisture content were calculated using formula shown below (Peavy, 1985).

$$M_c = \left[\frac{W_w - W_d}{W_w}\right] \times 100\% \tag{1}$$

Where,

M_c = Moisture content (%) of material
W_w = Wet weight of the sample
W_d = Weight of sample after drying.

Table 13 shows the result of moisture content of sample biomass after drying process under 105°C for one hour. Based on the table above, it clearly shows that EFB has lost a lot of moisture content during drying process compared to PMF and PKS.

Table 4-1: Moisture content of raw biomass

Type of sample	Weigh of sample Initial (g)	 Final (g)	 ∆ weight (g)	 MC %
EFB	2.0791	1.9328	0.1463	7.0366
PMF	2.0109	1.8397	0.1712	8.5136
PKS	2.0188	1.8081	0.2107	10.4368

After the torrefaction process, the moisture content of the torrefied biomass were calculated again. The result of moisture content of torrefied biomass is shown in Table 12 below.

Table 4-2: Moisture content of torrefied biomass

Type of sample	Weigh of sample Initial (g)	 Final (g)	 ∆ weight (g)	 MC %
EFB	2.5534	2.4912	0.0622	2.4356
PMF	2.9415	2.7650	0.1765	5.9980
PKS	2.8611	2.6058	0.2553	8.9208

Figure 4-2 shows the moisture content of raw and torrefied biomass. According to the Figure 4-2, the moisture content of the biomass decrease after the torrefaction process. EFB exhibits the largest loss in moisture content compared to the others.

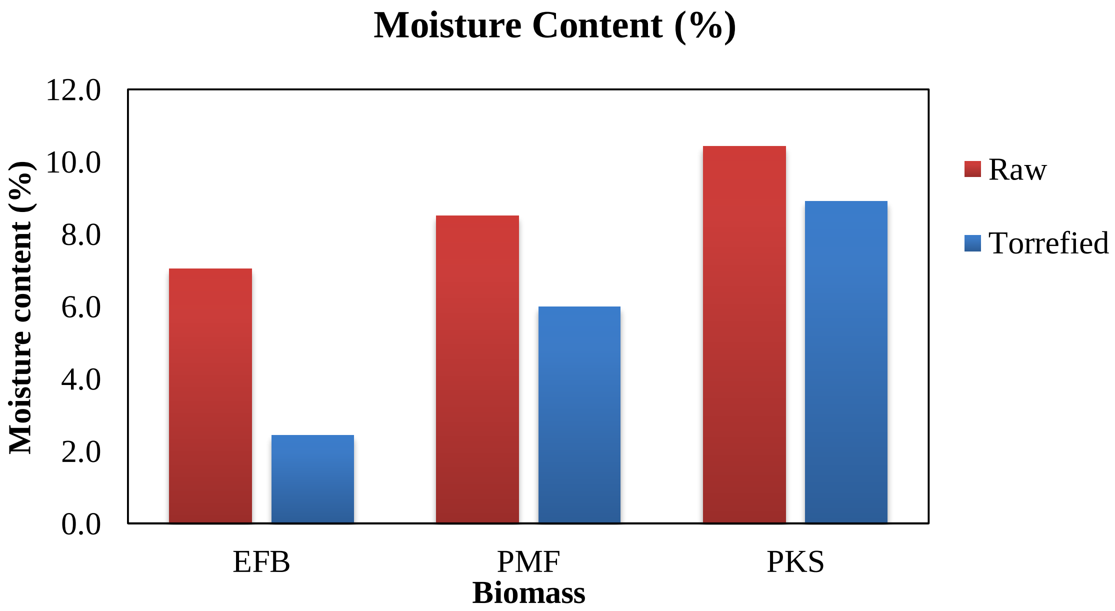


Figure 4-2: Moisture content of raw and torrefied biomass

4.4 *Yield of torrefaction*

The mass and energy yield of the torrefied biomass were calculated, based on the equation used by Bridgeman et al. The mass and energy yields (Y_{mass} and Y_{ene}) are defined by equations (2) and (3), respectively.

$$Y_{mass} = 100 \times (\frac{mass\ after\ drying\ or\ torrefaction}{mass\ of\ wet\ sample\ before\ treatment}) \qquad (2)$$

$$Y_{ene} = Y_{mass} \times \frac{HV\ after\ treatment}{HV\ before\ treatment} \times 100 \qquad (3)$$

Mass yield of the biomass is calculated by using equation (1) and the result of mass yield obtained is shown in Table 4-3 and Figure 4-3 below.

Table 4-3: Mass yield of raw and torrefied biomass

		EFB			PMF			PKS		
Temperature (ºC)		230	270	300	230	270	300	230	270	300
Mass (gm)	**Before**	2.0059	2.0064	2.0048	2.0064	2.0066	2.0068	2.0075	2.0053	2.0042
	After	1.3223	1.1323	1.0488	1.5945	1.4701	1.4344	1.8273	1.7654	1.6927
Mass Yield (%)		65.92	56.43	52.31	79.47	73.26	71.48	91.02	88.04	84.46

Based on Figure 4-3, it obviously shows that the mass yield of biomass decrease as the torrefaction temperature increase. Among three types of biomass, EFB demonstrates the lowest value for the mass yield while PKS has the highest value of mass yield. This means that EFB decomposed more than other two during the torrefaction. There are two main causes for the decrease in mass of the dried or torrefied samples. One is moisture loss, another thermal decomposition to form volatile of gases products such as CO, CO_2, and H_2O (Prins, et al., 2006). For the temperature 230 ºC samples, the decreasing value of mass is mainly caused by the loss of moisture. It means, substantial torrefaction did not occur for the biomass samples used in this study at 230 ºC. For torrefaction at higher temperatures (270 ºC and 300 ºC), the decrease is attributed to the thermal decomposition of hemicellulose part of the biomass (Ptasinski, et al., 2006).

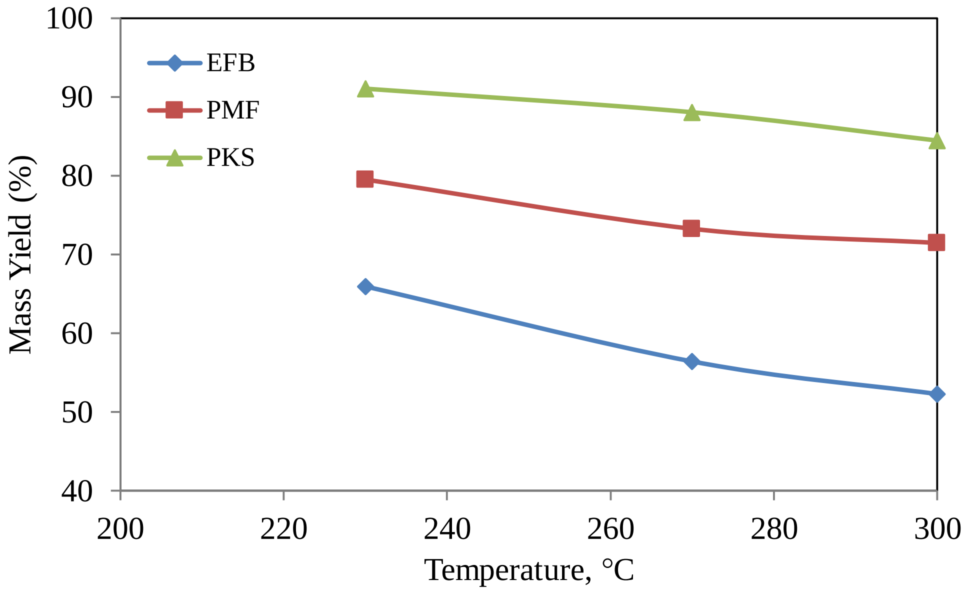


Figure 4-3: Mass yield of raw and torrefied biomass

The energy yield used by Bridgeman et al is in high heating value. The energy yield used in this study is in low heating value basis. Heating value is the amount produced by complete combustion of a unit quantity of fuel. It can be calculated using formula (4) below.

$$H_g = \frac{tW - e_1 - e_2 - e_3}{m} \tag{4}$$

Where,

t= temperature difference

W= 2409.26 cal/ºC

e_1= correction in calories for heat of formation of nitric acid

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 cm of fuse wire consume in firing)

m= mass of sample (0.5gm)

Correction in calories for heat of formation of nitric acid and sulphuric acid is neglected (Thermofluids Laboratory, 1999). The result for heating value for raw and torrefied biomass has been shown in Table 4-5. Based on the table below, roughly, the results indicate that, the heating value of biomass for PKS show the highest, followed by PMF and EFB. This result obtained because of the differences of chemical composition in each type of biomass. PKS comprise the highest chemical constituent like hemicellulose, cellulose and lignin.

Table 4-4: Heating value of raw and torrefied biomass

	EFB (MJ/kg)	**PMF (MJ/kg)**	**PKS (MJ/kg)**
Raw	18.0196	18.0462	22.0623
Torrefied at 230ºC	20.0668	22.0944	24.0321
Torrefied at 270ºC	24.0773	22.0867	24.0243
Torrefied at 300ºC	23.1178	23.8419	25.5686

Meanwhile, Table 4-5 and Figure 4-4 shows the result of energy yield of the torrefied biomass. The trend for energy yield of biomass is differ from each other. For EFB, the energy yield increase as the torrefaction temperature increase. Meanwhile, for PMF and PKS, both sample show the highest value of energy yield at temperature 230ºC and decrease gradually at temperature 270ºC. But, at 300ºC, the value of energy yield were increasing again. The differences of the value occur because the process is strongly depend on the decomposition temperature of the lignocellulosic constituent in biomass (Batidzirai , et al., 2013)

Table 4-5: Energy yield of torrefied biomass

	EFB (%)	**PMF (%)**	**PKS (%)**
Torrefied at 230ºC	73.41	97.29	99.15
Torrefied at 270ºC	75.41	89.67	95.86
Torrefied at 300ºC	75.83	94.43	97.88

When comparing between EFB, PMF and PKS, it is obvious that EFB shows the lowest value among the three. It means that EFB less decomposed than the other two during the torrefaction. The energy yield of torrefied PMF and PKS shows the highest value at the temperature 230 ºC and decrease at temperature 270 ºC. At 300 ºC, the energy yield increasing almost at 100%. However, the trend for EFB is totally difference from PMF and PKS. For EFB, the energy yield reveals the escalating as the temperature rise. But, the highest value of energy yield of EFB only 70 to 80% comparing to PMF and PKS that exhibit almost 90 to 100% of energy.

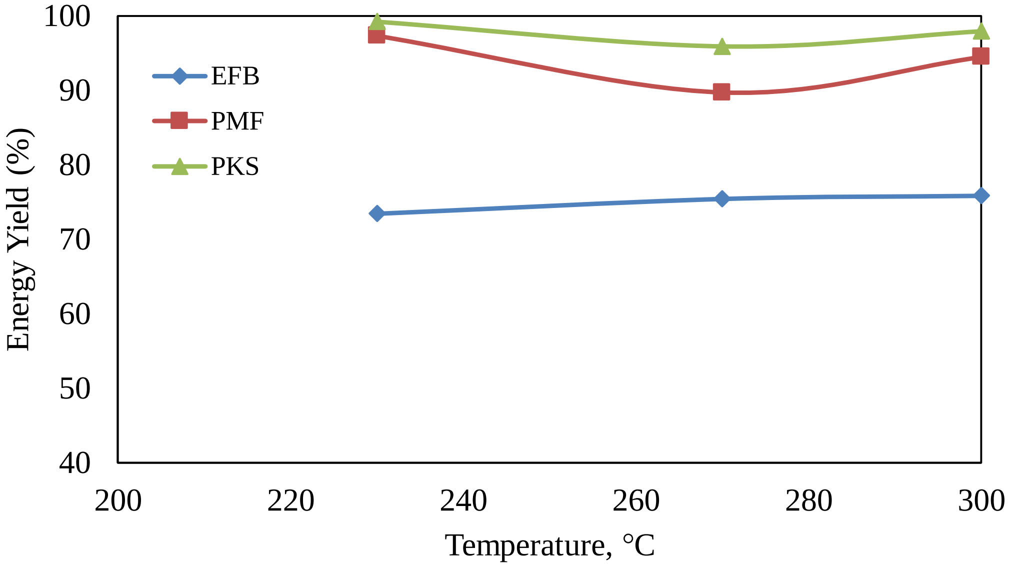


Figure 4-4: Energy yield of torrefied biomass

5 CONCLUSION

Based on the result obtained during the experiment, the PKS exhibit the highest heating value compare to EFB and PKS. Since the heating value of PKS at the highest value, it clearly shows that palm kernel shell (PKS) showed excellent energy yield values of 90% to 100% respectively. On the other hand, EFB exhibited a rather poor yield of 70%. Therefore, EFB is the least choice to be an alternative source to substitute the non-renewable energy source. As a recommendation, the experiment can be conducted using various types of palm oil waste such as fronds or trunks. Besides that, adding more parameter such as time, mass, and size distribution for the experiment. Lastly, adding more analysis of the raw and torrefied biomass such as chemical constituent in the biomass or volume of gas released during the experiment.

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