# PRODUCTION OF THE HIGH QUALITY TORREFIED OIL PALM BIOMASS FOR EFFICIENT ENERGY APPLICATION

TAN BAN LIN

BACHELOR OF CHEMICAL ENGINEERING UNIVERSITI MALAYSIA PAHANG

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# PRODUCTION OF THE HIGH QUALITY TORREFIED OIL PALM BIOMASS FOR EFFICIENT ENERGY APPLICATION

TAN BAN LIN

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

Faculty of Chemical & Natural Resources Engineering UNIVERSITI MALAYSIA PAHANG

JANUARY 2014

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# SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering (Biotechnology).

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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.

Signature:Name: TAN BAN LINID Number: KA10145Date: JANUARY 2014

# Dedication

Highest gratitude to my supervisors, my family members and my friends for all your care, support and trust on me. Special dedication to Faculty of Chemical and Natural Resources of University Malaysis Pahang on providing all the related environment and appropriate equipments on finishing my study.

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## ABSTRACT

The modern technology nowadays has led to a high consumption of energy where renewable energy sources become one of the hottest topics among the scientists. Biomass, as one of the promising renewable energy sources become the most attractive energy source in the world. Hence, this study aims to produce the high quality of torrefied oil palm biomass for efficient energy application. Torrefaction is known as the mild form of pyrolysis where the process occurs at atmospheric pressure and the temperature ranging of 473-573 K with the absence of oxygen. The raw materials selected for the study are the empty fruit bunch (EFB), mesocarp fiber and the kernel shell. The main reason of selected oil palm waste as the biomass raw material is due to its availability as Malaysia is the second largest producer of palm oil in the world. The experiment was carried out by using a tubular reactor which was located at the Gas Engineering Lab of University Malaysia Pahang. Results obtained showed that the powder form of all the samples have fully decomposed after torrefaction process, hence, the experiment was carried on with the fibrous form of samples for a better scale of study. The calorific value (CV) determined using a bomb calorimeter and the result collected showed a trend of increased in CV for all the samples started from EFB to kernel shell as the temperature increased. While for the case of mass yield, the trend was decreased for all the samples when the temperature increased along the torrefaction process. This was due to the thermal decomposition of the structure of samples during the experiment. This had directly affected the trend of energy yield by all the samples as the optimization of energy yield was not achieved at 100% except for kernel shell at 523K and 573K. Fourier Transform Infrared Spectroscopy (FTIR) test was also carried out in order to determine the component and chemical compositions which existed before and after the experiment, and results showed that the structure of the samples was mainly remain the same after the process.

Key words: torrefaction, oil palm biomass, calorific value, mass yield, energy yield, FTIR

## ABSTRAK

Zaman moden teknologi kini telah menyumbang kepada penggunaan tenaga yang melambung naik sehingga kajian terhadap sumber tenaga baru menjadi topik terhangat di kalangan scientist di dunia. Biomas, dikenali sebagai sumber tenaga boleh diperharui yang terpenting di dunia untuk dijadikan sebagai bahan api. Namun begitu, biomas masih mempunyai sesetengah kelemahan dari segi sifat-sifat kimia dan fizikal yang perlu ditangani untuk menjamin prestasi biomas sebagai sumber tenaga. Tujuan kajian ini ialah memajukan prestasi biomas sebagai sumber tenaga melalui proses torrefasi. Torrefasi dijalankan untuk menjamin kualiti biomas tanaman bijih timah dalam pembekalan sebagai sumber tenaga. Torrefasi merupakan satu proses yan berlaku pada tekanan atmosfera dalam suhu di lingkungan 473-573 K tanpa melibatkan kewujudan oksigen. Biomas tanaman bijih timah yang terpilih adalah EFB, mesocarp fiber dan kernel shell. Biomas tanaman bijih timah dipilih sebagai bahan mentah untuk kajian ini adalah disebabkan Malaysia merupakan pembekal bijih timah yang kedua terbesar di dunia dan ini dapat menjamin pembekalan biomass bijih timah yang berlanjutan. Eksperimen torrefasi dijalani dengan menggunakan reaktor tiub yang terdapat di makmal kejuruteraan gas yang bertempat di Universiti Malaysia Pahang. Merujuk kepada keputusan yang diperolehi, serbuk bahan mentah didapati mengurai sepenuhnya selepas proses torrefasi dan ini mengakibatkan tiada hasil keputusan untuk dianalisasi. Nilai kalori pula ditentukan dengan menggunakan bom kalorimeter dan keputusan menunjukkan nilai kalori kian meningkat untuk semua sample bahan mentah. Untuk kajian nilai hasil berat sample bahan mentah, trend menujukkan kejatuhan nilai hasil berat yang disebabkan oleh penguraian sample bahan mentah selepas proses torrefasi. Nilai hasil tenaga tidak mencapai keputusan yang sepatutnya kerana kerendahan nilai hasil berat bahan mentah menjejaskan nilai hasil tenaga yang sepatutnya. Manakala untuk kajian FTIR, keputusan menunjukkan struktur bahan mentah tidak mempunyai perbezaan yang ketara sebelum dan selepas proses torrefasi.

*Kata Kunci*: torrefasi, biomass tanaman bijih timah, nilai kalori, nilai hasil berat, nilai hasil tenaga, FTIR

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# LIST OF ABBREVIATIONS

CV	Calorific Value
FTIR	Fourier Transform Infrared
HHV	High Heat Value
МС	Moisture Content
MSW	Munipal Solid Waste
Ym	Mass Yield
Ye	Energy Yield

# **CHAPTER 1**

# INTRODUCTION

#### 1.1 Background

Fossil fuels are the primary energy source in the past and today's transportation fuel supply. However, the fossil fuels are not renewable and the applications of fossil fuels bring environmental problems which endangered the health of human being (Ren et al., 2012). Due to the worldwide environmental issues such as greenhouse effect, acid rain and global warming situation, the utilization of renewable energy source has consequently become increasingly of interest (Wannapeera & Worasuwannarak, 2012). The transition to a society driven by renewable energy sources such as solar, wind, biomass, tide, wave and geothermal energy next to energy savings becomes even more an important alternative in the field of energy consumption. According to the World Energy Outlook, renewable energy sources are expected to be the fastest growing energy sources. Biomass is known as the only renewable source that is based on sustainable carbon among this spectrum of several different energy sources (Stelt et al., 2011). Biomass acts as an important role in the future energy scenarios. This is due to its unique position as the only renewable source as sustainable carbon carrier caused it to be an attractive energy source. Unlike fossil fuels, biomass is planted and collected annually that can provide a continuous energy supply and hence is known as one of the most important and large renewable fuel sources (Ren et al., 2012). Biomass can be converted into energy via thermo chemical conversions, biochemical conversions and extraction of oil from oil bearing seeds (Stelt et al., 2011). However, biomass is classified as the low grade fuel due to its undesired properties such as high moisture content, high ash content and low energy density. Therefore, direct utilization of biomass seems to face great obstacles in overcoming the above drawbacks. (Wannapeera & Worasuwannarak, 2012).

In order to overcome the undesired properties of biomass, many experiment methods have been carried out such as pelletisation, gasification, pyrolysis or torrefaction process, hence this lead to this study on torrefaction process. As mentioned in the study of Bergman (2005), densification by means of pelletisation is considered to be the proven technology to improve biomass properties for its conversion into heat and power. The biopellets market is becoming quite mature with serious outlets in the domestic market and the energy market after a significant increase of pellets production in Europe and Northern America. It is believe that biopellets are a major sustainable fuel to replace coal. While for the gasification, it is the partial oxidation of carbonaceous feedstock above 800 °C to produce a syn-gas that can be used for many applications such as gas turbines, engines, fuel cells, producing methanol and hydrocarbon. It is desirable that gasification becomes increasingly applied in future rather than direct combustion due to its higher efficiency. It is believed that coupling gasification with power systems increases the efficient use of thermal energy streams (Stelt et al., 2001). Pyrolysis is a thermal chemical technology conducted at the range of temperature of 400-600°C in the absence of oxygen. However, pyrolysis biomass is normally heated and decomposed to produce liquid biomass such as hydrocarbon biofuel (Ren et al., 2012). In order to obtain solid wastes as products, torrefaction process is the preferable method. According to Bergman (2005), torrefaction is a thermal chemical treatment of biomass at 200-300°C which is carried out under atmospheric conditions and the absence of oxygen. The techniques is carried out under a relatively low range of temperature (200-300°C) which aims to enhance the fuel properties attractively for further utilization such as combustion, gasification and co-combustion (Bergman, 2005).

## **1.2 Motivation**

Malaysia as a tropical country experiences hot and wet weather throughout the year. Besides that, Malaysia is with abunbant and relatively cheap supply of conventional fossil energy resources such as oil, gas, and coal as well as renewable energy sources such as hydropower, biomass and solar. According to Yusoff (2004), past and current economic growth in the country has been primarily fueled by fossil fuels. However, recently, the economic recovery upward trend combined with recent strategies to minimize the cost as much as possible has developed a supportive environment to incorporate energy conservation and energy efficiency measure as part of the nation's "Vision 2020" industrialization objectives. This also in line with the primary national energy policy objectives of the country, under its utilization objective, namely, to promote and encourage the efficient utilization of energy as well as discourage wasteful and unproductive patterns of energy consumption. Hence, this is lead to the study of this process. Besides that, over the last few decades, the Malaysian palm oil industry has grown to become a very important agriculture-based industry, where the country is today one of the world's leading producer and exporter of palm oil. Malaysia as

the second largest producer of palm oil which come after Indonesia, covered 41% of the total world supply ensure a continuous supply of oil palm waste which been chosen as the raw materials of biomass for this study. Due to its availability in Malaysia, oil palm waste are considered as the best among all biomass waste and this directly lead to the reason why oil palm waste as torrefied biomass is the topic of this study. As can been seen previously, most of the torrefaction biomass process are emphasized on the woody mass such as the TOP process by Bergman (2005) and the upgrading of woody biomass by torrefaction of Wannapeera & Worasuwannarak (2012). Besides that, according to Masuda et al., 2001, the amount of oil palm solid wastes sums up to 26 million tonnes per year, which is about three times as large as the total amount of plastics discarded in Japan annually. Hence, it is necessary to carry out more study on the other raw materials of biomass instead of woody biomass especially oil palm wastes. A full utilization of oil palm waste as biomass will ensure an economical advantage towards the development of Malaysia in the field of renewable source, meanwhile, reduce the rate of environmental issues especially the carbon emission to the environment. Moreover, oil palm is a perennial tree crop, which is cultivated extensively in the humid tropical land. Average planting cycle of a palm tree is about 25 years for efficient productivity. This is due to the conversion of solar radiation to plant growth by photosynthesis, the chemical energy content of the harvested palm fruit and biomass exceeds the energy input through the farming system. These palm residues contain high nutrient value. Based on the nutrient content estimation by Yusoff (2004), an equivalent energy of 683.2 MJ is saved from the production of chemical fertilizer if the palm residues are used as fertilizer.

#### **1.3 Problem Statement**

Biomass is known as the only renewable source that is based on sustainable carbon among this spectrum of several different energy sources (Stelt et al., 2011). However, it still be treated as a low grade fossil fuel due to its undesirable properties. Hence, many researches have been carried out in improving the properties of biomass which can enhance its performance as fossil fuel. Torrefaction process has been selected as the main process of study in this research. Torrefaction is widely known as a thermal chemical treatment process of biomass which is carried out under the atmospheric pressure with the temperature range of 200-300 °C with the absence of oxygen (Bergman, 2005). It is also known as a mild form of pyrolysis, and is preferred compare to slow and fast pyrolysis due to the reason of the biomass used in this study is oil palm solid waste materials. Pyrolysis biomass is normally

heated and decomposed to produce liquid biomass such as hydrocarbon biofuel (Ren et al., 2012). On top of that, the selected biomass material for this study is the oil palm solid waste materials which are the empty fruit bunch (EFB), mesocarp fiber, and kernel shell. All of this solid biomass will undergo the torrefaction process and consequently, torrefied biomass is obtained. Research showed that the torrefaction process successfully overcome the undesired properties of biomass. It was stated that torrefaction process was found to be effective for improving the energy density and shelf life of the biomass (Uemura et al., 2011).

## 1.4 Objectives

This study aims to produce the high quality of torrefied oil palm biomass for efficient energy application.

#### 1.5 Scope

The study emphasize on the torrefaction of oil palm wastes as biomass where two main parameters will be tested in the study as shown below.

- Effect of different temperature ranging within 200-300°C.
- Effect of total surface area of biomass particles (Fibrous and Powder form).

The two parameters mentioned will be used to test on the moisture content and calorific value (CV) which contribute to the calculation of mass yield and energy yield. Hence, the characteristics of oil palm waste before and after torrefaction will be discuss in order to achieve the purpose of choosing torrefied oil palm biomass for energy application. The range of temperature set will be around 200-300°C, where graphs will be plotted accordingly to the result obtained. In this study, the oil palm solid wastes included are the empty fruit bunches (EFB), mesocarp fibre, and palm kernel shell. Once the experiment carried on, the properties of each raw material were characterized for their calorific value, moisture content, mass yield and energy yield. The experiment will be conducted in a pyrolyzer which is a horizontal tubular type reactor by differing the temperature in the ranging of 200-300°C located at the gas engineering lab in the FKKSA Laboratory of University Malaysia Pahang. While for the characterization of moisture content will be carry out by using an electric oven which located in the environmental engineering lab at the temperature of 105°C for duration of one hour. The calorific value is tested by using the bomb calorimeter which located at the basic engineering lab of UMP. CV ratio will be calculated based on the calorific value obtained in order to determine the energy yield of the study. Lastly, FTIR analysis will be carried out to

determine the bonds exist before and after the torrefaction process which occurred at different values of temperature. The analysis was carried out by using the spectrometer located at Faculty of Industrial Science and Technology of University Malaysia Pahang.

## 1.6 Structure of Thesis

Chapter 2 mainly discussed on the information gather and the review done by this study towards the topic concerned. It started with the review of biomass and types of biomass in the world, and is followed by the review of palm oil industry in Malaysia together with the palm oil solid wastes used in this study. Last part of this chapter was done on the biomass conversion techniques and methods used in industry. Meanwhile, comparison between the recent literature and pass researches also have been done in order to get a better review.

Chapter 3 was discussed on the samples and methods used along the experiment. Hence, methods used to characterize moisture content, calorifiv value, FTIR analysis and reactor used to run the torrefaction process were discussed and presented. Besides that, measurements used for the calculations of mass and energy yield was also discussed in this chapter.

Chapter 4 presented on the preliminary results and the final results obtained before and after the experiment. Hence, the effects of temperature on the mass and energy yields was discussed within the chapter. The results obtained was used to compare with the pass researches in this chapter. Lastly, discussion on FTIR analysis was done based on the condition before and after torrefaction process.

# **CHAPTER 2**

# LITERATURE REVIEW

### 2.1 Chapter Overview

Palm oil industries in Malaysia generate about 90 million tonnes of renewable biomass (trunks, fronds, shells, palm press fiber and empty fruit bunches) per year, including about 1.3 million tonnes of oil palm trunks, 8 million tonnes of pruned and felled fronds, and 2.4 million tonnes of oil palm empty fruit bunches (EFB) (Alam et al., 2009). Hence, it is necessary to carry out more researches in order to make full use of all the oil palm solid waste produced throughout the year.

In this chapter, the research done related to the topic will be studied and reviewed especially the researches on different type of process which have been carried out to enhance the properties of oil palm solid waste. Meanwhile, the study on the properties of all the oil palm solid waste included will be reviewed as well.

## 2.2 Biomass and Types of Biomass

Biomass is any organic matter such as wood, crops, seaweed, animal wastes that can be used as an energy source, hence biomass can be understood as regenerative (renewable) organic material that can be used to produce energy (Oyemakinwa, 2011). For thousands of years, people have burned wood to heat their home and cook their food. These are the most basic examples of using biomass as an energy supply. As stated in the NEED project of Secondary Energy Infobook 2012, biomass is probably the oldest source of energy after the sun. Biomass gets its energy from the sun as all the organic matter contains stored energy from the sun through a process called photosynthesis. Along the process, sunlight gives the plants the energy that they need to convert water and carbon dioxide into oxygen and sugars. These sugars are called carbohydrates which supply the plants and animals that eat plants with energy. Consequently, biomass is known as a renewable energy source because it supplies are not limited due the reason of trees and crops can always grow and waste will always exist (Baumann et al., 2012). Figure 2.1 below showed the process of photosynthesis by the plant.



Figure 0-1: Process of Photosynthesis in Plant (Baumann et al., 2012)

Producing fuel and energy from biomass is a complex procedure but the principle behind it corresponds directly to the process of photosynthesis. In layman's term, that means biomass is manufactured from the wood and agricultural products, food waste, landfill gas and biogas, and lastly alcohol fuel (Oyemakinwa, 2011). The types of the following types of biomass will be further discussed in the subchapters below.

## 2.2.1 Woody Biomass

Woody biomass is best described as the material obtained from trees or the products of tress that has accumulated to a sufficient quantity that is a hazard of disposal problem or from trees specifically managed for biomass markets (Shelly, 2011). The previous definition also precludes wood and wood residue that would otherwise fit the definition except that they are already used to produce higher-value products, such as sawmill residues for particle board and other composite panels. Woody biomass is the solid portion of stems and branches from trees or residues products made from trees. Woody biomass can come from variety sources, including:

• Non-timber tree removal - removing dead and dying trees, unwanted urban trees or trees impeding land development.

- Forest management harvesting the removal of small diameter trees from overpopulated stands for wildfire hazard fuel reduction, pre-commercial thinning of timber stands or forest health improvement.
- Timber harvesting and logging residues non-merchantable wood including branches, undersized trees and non-commercial species removed during typical timber harvesting operations.
- Sawmill and other wood manufacturing residues includes bark, undersized and defective wood pieces, sawdust and other wood waste.
- Landfill diversion wood debris from tree removal and pruning, construction, demolition, discarded shipping materials and other trashed wood products.
- Chaparral management removal of excess woody shrubs and plants for wildfire fuel hazard reduction or other vegetation management goals.

Woody biomass utilization is sometimes narrowly defined to mean the use of wood as a soure or feedstock solely for the production of energy (heat and electricity). This is short-

sighted and often hinders the discovery of its full potential. It is important to remember that al though woody biomass is low in value and quality it has potential as a feedstock for energy pr oduction as well as for higher value manufactured goods (Shelly, 2011).

## 2.2.2 Municipal Solid Waste (MSW) as Biomass

Municipal solid waste (MSW) is defined as household waste, commercial solid waste, nonhazardous sludge, conditionally exempt, small quantity hazardous waste, and industrial solid waste. MSW includes food waste, rubbish from residential areas, commercial and industrial wastes, and construction and demolition debris. Biosolids which is the byproducts from waste water treatment, also know as sewage sludge are not included in the formal definition of municipal solid waste, though it is estimated that 20% of dry short tons of biosolids generated annually are handled by MSW landfills (Valkenburg et al., 2008).

The composition of MSW is mainly made up by the following materials,

- Paper or paperboard
- Food Scraps
- Wood
- Yard trimmings
- Plastics

- Metals
- Rubber, leather and textiles
- Glass

The origin of a given material can be very diverse and this complicates the picture. Some items, such as fridges or computers, are intricate machinery made of numerous components and many different materials (plastic, glass, metal, etc). Consequently, other categorising systems could be chosen such as listing of durable/non-durable goods or combustible/non-combustible matter. The classification system can and should be designed according to the need of its user. It is obvious that MSW is a complex and heterogenous mixture, made of materials with very different chemical structure and physical properties. However, a further obstacle is appearing: the category "other". This category is far from minute and may represent a significant share of the total MSW amount and can therefore make difficulties for waste management handling (Becidan, 2007).

#### 2.2.3 Landfill Gas and Biogas as Biomass

Bacteria and fungi are not picky eaters. They eat dead plants and animals, causing them to rot or decay. A fungus on a rotting log is converting cellulose to sugars to feed itself. Although this process is slowed in a landfill, a substance called methane gas is still produced as the waste decays. New regulations require landfills to collect methane gas for safety and environmental reasons (Baumann et al., 2012).

Methane gas is colorless and odorless, but it is not harmless. The gas can cause fires or explosions if it seeps into nearby homes and is ignited. Landfills can collect the methane gas, purify it, and use it as fuel. Methane, the main ingredient in natural gas, is a good energy source. Most gas furnaces and stoves use methane supplied by utility companies.

In 2003, East Kentucky Power Cooperative began recovering methane from three landfills. The utility now uses the gas at six landfills to generate enough electricity to power about 9,000 Kentucky homes. Today, a small portion of landfill gas is used to provide energy. Most is burned off at the landfill. With today's low natural gas prices, this higher-priced biogas is rarely economical to collect. Methane, however, is a more powerful greenhouse gas than carbon dioxide. It better to burn landfill methane and change it into carbon dioxide than release it into the atmosphere. Methane can also be produced using energy from agricultural and human wastes. Biogas digesters are airtight containers or pits lined with steel or bricks.

Waste put into the containers is fermented without oxygen to produce a methane-rich gas. This gas can be used to produce electricity, or for cooking and lighting. It is a safe and cleanburning gas, producing little carbon monoxide and no smoke. Biogas digesters are inexpensive to build and maintain. They can be built as family-sized or community-sized units. They need moderate temperatures and moisture for the fermentation process to occur. For developing countries, biogas digesters may be one of the best answers to many of their energy needs. They can help reverse the rampant deforestation caused by wood-burning, reduce air pollution, fertilize over-used fields, and produce clean, safe energy for rural communities (Baumann et al., 2012).

#### 2.2.4 Use of Biomass as Energy

The wood is usually burned in order to use the energy for heating purpose. However, burning is not the only way to convert biomass energy into a usable energy source. There are four ways, started with the process of fermentation which is a process of producing alcohol from various plant especially corn. The two most commonly used processes involve using yeast to ferment the starch in the plant to produce ethanol. One of the newest processes involves using enzyme to break down the cellulose in the plant fibers, allowing more ethanol to be made from each plant, because all of the plant tissue is utilized but not just the starch (Baumann et al., 2012). Bacterial decay is another method of producing methane by feeding bacteria on dead plants and animals. Methane is produced whenever organic material decays. Methane is the main ingredient in natural gas, the gas sold by natural gas utilities. Many landfills are recovering and using the methane gas produced by the garbage. Lastly, biomass can be converted into gas or liquid fuels by using chemicals or heat. In India, cow manure is converted to methane gas to produce electricity. Methane gas can also be converted to methanol, a liquid form of methane (Baumann et al., 2012).

In conclusion, biomass is considered as one of the most promising renewable energy sources in the world nowadays. It is utilized as solid, liquid or gas fuel which opts in the field of renewable source of fossil fuel. Especially, lignocellulosic biomass wastes are attracting interest worldwide, because of its non-edible characteristic (Uemura et al., 2011). The nonrenewable fossil oils and the application of fossil oils brings environmental problems, especially the carbon dioxide emission in contributes to the issue of global warming. Consequently, these problems motivate scientists and researchers to look for the renewable sources and here comes biomass. The availability of biomass due to its plantation and collected annually ensures a continuous energy supply (Ren et al., 2012). In order to utilize biomass waste efficiently, the following drawbacks about biomass compared to fossil fuels must be solved properly, higher energy consumption for collection, heterogeneous and uneven composition, lower calorific value and difficulty in transportation (Uemura et al., 2011).

## 2.3 Palm Oil Industry in Malaysia

Palm oil is an agricultural product, which is mainly produced in South-East Asian countries, especially Indonesia and Malaysia. Oil palm waste is generated through the production of palm oil. While in Malaysia, the climate of hot and wet weather throughout the year encourages the growth of the oil palm and consequently the development of oil palm cultivation in Malaysia. This has lead Malaysia as a major global oil palm biomass producer and a main exporter in the world. The total oil palm planted area in Malaysia reached 4.98 Mha as of September 2011 which covers approximately 73% of the agricultural land and makes oil palm a promising raw material for renewable energy generation (Ng et al., 2012).

In the Eight Malaysia Plan in 2001, renewable energy was introduced as the 'fifth fuel' after the four energy sources: oil, gas, hydropower and coal. The fifth fuel has been gaining influence in current energy development as a potential alternative to fossil fuels (National Energy Policies , 2006). According to the study by Sulaiman et al. (2011), energy consumption in Malaysia has been increasing since 1994. The final commercial energy demand by source for the years 2000-2010 is presented in the Table 2.1 (Sulaiman et al., 2011).

Table 2-1: Final commercial energy demand by source for the years 2000-2010 (Sulaiman et al., 2011)

	Average En (MW)	nergy Dema	and	%	of Total	
Source	2000	2005	2010	2000	2005	2010
<b>Petroleum Products</b>	26002	32442	43534	65.9	62.7	61.9
Natural Gas	5131	7820	11098	13	15.1	15.8
Electricity	6989	9830	13318	17.7	19	18.9
Coal and Coke	1316	1649	2378	3.4	3.2	3.4
Total	39437	51741	70329	100	100	100

As can be seen, Malaysia is highly dependent on fossil oil as an energy source. Malaysia is working towards fuel diversification to reduce its dependency on fossil fuels in order to improve the energy security. Palm biomass appears to be one of the potential energy sources due to its abundance. In addition, the realisation of palm biomass for producing value-added products and biochemical increases the business opportunities for the palm biomass industry. The industry is forecasted to evolve as a major sector in Malaysia's future development. Green development indicators are of the utmost importance in ensuring economic and sustainable development (Sulaiman et al., 2011).

Despite the large amount of palm oil production, the oil contributes to less than 25% by weight of the palm fruit bunch (FAO, 2011). For every kg of palm oil produced, approximately four kg of dry biomass is produced, excluding palm oil mill effluent (POME). In 2010, 88.74 Mt of Fresh Fruit Bunch (FFB) of oil palm was processed (GGS, 2011). The amount of biomass available from the stated is listed in the Table 2.3(b). 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 (GGS, 2011). The potential energy that can be generated is calculated in Table 2.3(b) and totals up to 37 Mt/y of oil equivalent based on the amount of biomass available as of 2010. This amount of energy may be wasted due to the inefficient utilisation of the available palm biomass. To date, 60 MW out of 68 MW of biomass power is generated from palm biomass. The government of Malaysia has set a target to increase its biomass power generation capacity to 800 MW by 2020, and 500 MW is to be generated from palm biomass (Kementerian Tenaga, 2011). The cumulative renewable energy target on biomass projected by the Ministry of Energy, Green Technology and Water (Kementerian Tenaga, 2011) is illustrated in Figure 2.2.

Quantity (Mt/y)	Net Calorific Value (MJ/t)
21.27	18795
10.8	19055
4.98	20093
49.85	16992
86.9	
	21.27 10.8 4.98 49.85

Table 2-2: Potential Energy that can be generated (Sulaiman et al., 2011)



Figure 2-2: Cumulative Renewable Energy target on Biomass Projected (Sulaiman et al., 2011)

### 2.4 Palm Oil Solid Wastes



Figure 2-3: Fresh oil palm fruit and its longitudinal section (Guo & Lua, 2001)

Figure 2.3 showed a fresh oil palm fruit and its longitudinal section. During palm oil plantation and processing, a large amount of solid wastes such as palm trunks, palm fronds, empty fruit bunches (EFB) and fruit wastes which including the extracted mesocarp fibers and palm shells, are generated (Guo & Lua, 2001). In Malaysia, which is one of the largest palm oil producer in the world, about 2 million tons (dry weight) of palm shells and 1 million tons of extracted fibers are estimated to be produced annually. Normally, these waste are used as boiler fuel or chemical feedstock for solid (char), liquid (aqueous and tar fractions) and gaseous products (Guo & Lua, 2001). Figure 2.4 shows the process flow diagram of a palm oil mill where to obtain the waste needed in the study which is EFB, mesocarp fiber and kernel shell.



Figure 2-4: Process flow diagram of palm oil mill (Uemura et al., 2011)

## 2.4.1 Empty Fruit Bunches (EFB)

According to Singh et al., 2010, **a**n average oil palm mill can handle about 100 metric tonnes (mt) of fresh fruit bunches daily. Solid residues and liquid wastes are generated at the mills where oil extraction takes place. The solid residues, mainly EFB, are more than 20% of the fresh fruit weight. The EFBs are either incinerated or applied to fields. These practices create environmental pollution problems such as incineration and boilers emits gases with particulates such as tar and soot droplets of 20-100 microns and a dust load of about 3000-4000 mg/nm and indiscriminate dumping of EFB causes additional methane emission into the atmosphere (Amal et al., 2008). A new usage for these wastes ought to be looked into in order to minimize the pollution.

EFB is a suitable renewable raw material for bioconversion into value added products because it is easily accessible, abundant locally and rich in lignocellulose as shown in Table 2.3 below.

Components	Sreekala et al. (1997)	Khalil et al. (2007)
Lignin	25-35	21.2
Cellulose	45-50	49.6
Hemicellulose	25-35	18
Ash	-	2

Table 2-3: Fibrous composition of major constituents in EFB (%) (Mohammaad et al., 2011)

EFB was often used as fuel to generate steam at the palm oil mills. However, the air pollution from EFB burning caused serious environmental concerns and the authorities formulated tight regulatory controls to curb air-pollution from such activities. EFB is now mainly used as mulch in the palm oil plantations to control weeds, prevent erosion and maintain soil moisture. However, due to escalating labor, transportation and distribution costs of EFB in the field, its utilization as mulch is becoming more expensive. There is a growing interest in the low and cost attractive solid state bioconversion of EFB into value added products, such as compost, citric acid or enzymes (Alam et al., 2009). Table 2.4 showed the approximate compositions of major constituents in EFB.

Table 2-4: Approximate compositions of major constituents in EFB (%) (Mohammad et al., 2011)

Nutrients	Amal et al. (2008)	Suhaimi and Ong (2001)	Hajar (2006)	Rozainee et al. (2001)
С	48.8	43.7	42-43	50.09
Nutrients	0.2	0.52	0.65-0.94	2.05
C.N	-	-	45-64	-
Р	-	0.05	-	-
Н	6.3	-	-	7.16
0	36.7	-	-	40.16
K	-	1.34	-	-
S	0.2	0.07	-	0.06
В	-	4	-	-
CA	-	0.19	-	-
CU	-	13	-	-
Mn	-	-	-	-
Mg	-	20	-	-
Zn	-	21	-	-

Fe	-	649	-	-	
Ash	7.3	-	4.8-8.7	5.74	
Oil	-	-	8.1-9.4	-	
P2O2	-	-	0.18-0.27	-	
K2O	-	-	2.0-3.9	-	
MgO	-	-	0.25-0.4	-	
CaO	-	-	0.15-0.48	-	

Besides that, the EFB fiber is coarser and stronger than that obtained from the pericarp. By adding a binding agent, such as rubber latex, the EFB fiber can be used for cushion filling material. One of the most promising products manufactured from the EFB is the medium density fiber (MDF) board which will be on the production line as soon as a factory is set up. The EFB has a great potential in these applications. It should be noted that products like coir fiber, fiber board, cement board, roofing tile and card paper can be produced from the EFB or fiber (Prasertsan & Prasertsan, 1996).

#### 2.4.2 Mesocarp Fiber

Palm-pressed fiber or mesocarp fiber constitutes about 15.7 wt% of the solid biomass of fresh fruit bunches (FFB). It is an elongated cellulose material with 30-50 mm length which has been found to trap about 5-7 wt% of residue oil after the extraction of palm oil mill. Conventionally, the fiber is mixed with kernel shell and being utilized as solid fuel to generate electricity for the mill while the excess fiber and empty fruit bunch are the transported to the plantation for the field mulching (Lau et al., 2008). It is known that palm-pressed fiber oil is enriched with natural carotene, vitamin E, sterols, squalene, co-enzyme  $Q_{10}$  and phenolic compounds (Choo et al., 1996). These functional components have been determined to possess certain biological activities such as  $\beta$ -carotene having antioxidant activity and inhibiting growth of colon cancer cells; lycopene is particularly an effective singlet oxygen quencher; vitamin E (e.g., tocotrienols) as antioxidant, anti-cancer and having hypercholesterolemic effects; squalene as chemopreventive agent against some type of cancers, coenzyme  $Q_{10}$  with protective effect against artherosclerosis and heart disease, and lastly phenolic components with superior antioxidant property (Lau et al., 2008).

Besides that, the oil retained in its cell wall makes the mesocarp fiber a good combustible material. In factories which produce steam and electrical power, all of the mesocarp fiber is used. However, only 30% is consumed if power is not produced. Therefore, in some

factories, about 70% of mesocarp fiber is considered as waste. Its ash contains 1.7-6.6% of P, 17-25% of K and 7% of Ca. It could therefore be used as source of minerals for plants. Although mesocarp fiber is similar to rice straw, it contains a higher percentage of fiber and lignin which cannot be digested easily by animals. However, its similarity to rice straw makes it a good substrate for mushroom cultivation. The interesting part was mesocarp fiber is suitable for the pulp and paper industry as well (Prasertsan & Prasertsan, 1996).

#### 2.4.3 Kernel Shell

Palm kernel shell covered about 8 wt% of the solid biomass of FFB. It is the most difficult waste to decompose compare to the mesocarp fiber and EFB. The shell size is uniform and is not as bulky as the EFB. Kernel shell usually left unused in the factory or disposed by the land-fill method. In terms of energy, it is an energy intensive substance. However, if the conventional fuel is to replace by the kernel shell, substantial modification of the furnaces is needed and hence, many factories are still reluctant to use kernel shell unless they are economically forced to do so (Prasertsan & Prasertsan, 1996).

However, there is a possibility that the kernel shell can be used for activated carbon production or charcoal. It contains 20.3% of fixed carbon and is physically similar to the coconut shell, which has been used to produce the activated carbon successfully. It is anticipated that the stringent environment control measures will increase the demand for activated carbon in the future. It is possible that activated carbon can be applied for the decolorization of the unacceptably dark colored effluent of the palm oil mills. Hence, some factories have shown an interest in incorporating activated carbon production in the milling operation (Prasertsan & Prasertsan, 1996). Many mills have installed co-generation plants, to generate both heat and electrical power. High pressure steam passes through a back-pressure steam turbine to generate electricity sufficient for the mill consumption. The exhausted steam is used as heat source for the milling process. Only the fiber and sometimes a small amount of shell is fed into the boiler furnace. The heating value of the shell is 17.4 MJ/kg. It is appropriate to assume that the overall thermal efficiency of the co-generation system is 25%. In practice, if the kernel shell is used for electricity generation, 16 small co-generation systems have to be installed (Prasertsan & Prasertsan, 1996). Table 2.5 below showed the result and literature study of the physical properties of EFB, mesocarp fiber and kernel shell.

Biomass	Moisture (MJ/kg)	Calorific Value		Elementary and Ash Analyses (wt %)					
		Dry (HHV)	Dry (LHV)	С	Η	Ν	S	0	Ash
EFB (Study)	57.2	17.02	15.82	45.53	5.46	0.45	0.044	43.4	5.12
EFB (Literature)	65	19.1		48.8	6.3	0.2	0.2	36.7	7.3
Mesocarp Fiber (Study)	37.2	19.61	18.31	46.92	5.89	1.12	0.089	42.66	3.32
Mesocarp Fiber (Literature)	42	18.8		47.22	6	1.4	0.3	36.7	8.4
Kernel Shell (Study)	21.4	19.78	18.49	46.68	5.86	1.01	0.06	42.01	4.38
Kernel Shell (Literature)	17	20.1		52.4	6.3	0.6	0.2	37.3	3.2

Table 2-5: Biomass sample used and the physical properties (Uemura et al., 2011)

## 2.5 Biomass Conversion Techniques and Methods

Biomass can be converted into useful forms of energy using a number of different processes. Choice of conversion process depends on the type, property and quantity of biomass feedstock, the desired form of the energy, for example, the use requirements, environmental standards, economic conditions and project-specific factors. Biomass can be converted into three main products which are power or heat generation, transportation fuels and chemical feedstock (Saxena et al., 2007). Before proceeding to the main process of the study which is torrefaction, it is necessary to review the others method of biomass conversion. The biomass conversion method basically divided into two main common methods as shown below.

- Biochemical Conversion Process
- Thermochemical Conversion Process.

The significant difference or the characteristics of the two methods will be reviewed accordingly in the following passage.

### 2.5.1 Biochemical Conversion Process

Biochemical conversion process involved the application of chemicals and biological organisms such as bacteria, fungi, and enzymes to act on the material so as to produce some toxic materials to enhance its decomposition decay (Oyemakinwa, 2011). It is the process by

which biomass is converted to gas ( $CO_2/CH_4$ ), waste (compost or fertilizer) and water (water or  $C_2H_5OH$ ) by using microorganisms (Kucuk & Dermirbas, 1995). Biochemical is a nonpollution method, characterized by low energy consumption, has been studied by specialist mainly with regard to biogas, ethyl alcohol, compost and protein obtainment. Yet, the worldwide application of such procedures has not gone beyond preliminary experiments on a pilot scale, with few industrial results or as stations of producing biogas and compost, placed according to specific and local responsibilities (Kucuk & Dermirbas, 1995). Biochemical systems are among the most promising, environmentally sustainable alternatives for reducing atmospheric carbon dioxide levels. Biomass can act as a reservoir of carbon or as a direct substitution for fossil fuel with no net contribution to atmospheric  $CO_2$  if produced and used sustainably (Oyemakinwa, 2011). Below will be some example of biochemical processes which is widely used in industry.

#### 2.5.1.1 Anaerobic Fermentation

Anaerobic fermentation, where the waste is kept without oxygen for approximately 2-8 weeks at the temperature around 310K, not only solves the pollution problem but also produces energy and organic fertilizer from a renewable source. Animal waste has created a major waste disposal problem and is becoming more acute because greater numbers of animals are being raised on concentrated feedstock. The capacity of the local environment to dilute, stabilize and dissipate the accumulation of wastes from these systems is exceeded in many locations. Improper disposal of these wastes, such as spreading on the land, burying, burning or simply collecting in piles, pollutes the air and water streams, thereby creating a health hazard. During fermentation, gas is produced. The gas composition is 65-70% methane, 35-30% carbon dioxide and negligible traces of other gases (e.g. H<sub>2</sub>S and H<sub>2</sub>) and is saturated with water. The gas has an approximate heating value of about 26 MJ/m<sup>3</sup> (Kucuk & Dermirbas, 1995).

#### 2.5.1.2 Biological Hydrogen Production

According to recent literature of Saxena (2007), hydrogen will be an important energy carrier in the near future. Instead of fossil fuel, hydrogen production from biomass has to be employed as it is sustainable and renewable. All processes of biological hydrogen production are dependent on the presence of hydrogen-producing enzymes. It is hypothetically possible that the quantity or inherent activity of these enzymes could limit the overall process. It was found that all the enzymes contain complex metallocluster as active sites and that the active sites of the enzyme units are synthesize in a complex process involving auxiliary enzymes and protein maturation steps. Three enzymes that were found to carry out these reactions are nitrogenase, Fe-hydrogenase and NiFe hydrogenase (Saxena et al., 2007).

#### 2.5.1.3 Anaerobic Bioconversion

Anaerobic bioconversion of lignocellulosic material into methane and carbon dioxide is performed by a number of microorganisms in several stages: hydrolytic, acidogenic, homoacidogenic and methanogenic. The last one is a very important stage and plays a significant role in bioconversion. The activity of methanogenic bacteria, which are able to convert organic acids into methane and carbon dioxide, depends on several factors such as temperature, pH, concentration of substrates and minerals. For methanogenic bacteria, pH ranges will be between 6.6 and 7.6 whereas the maximum range of pH was observed between 7.2 and 7.6. However, anaerobic digestion of cellulosic materials in digesters is a slow process, mainly due to the lignin. On the contrary, some natural anaerobic ecosystems are efficient in degrading lignocellulosic materials. Typical examples are the fore stomachs of ruminants. Rumens microorganisms have been shown to be capable of converting a wide range of lignocellulosic material into biogas in a two phase rumen derived process with efficient in the range of 50-60% (Kucuk & Dermirbas, 1995).

### 2.5.2 Thermochemical Conversion Process

The processes under thermochemical conversion might not really produce useful energy directly, but under controlled temperature and oxygen conditions, it can be used to convert the original biomass feedstock into more convenient forms of energy carriers, such as producer gas, oils or methanol. These carriers are either more energy dense and therefore reduce transport costs, or have more predictable and convenient combustion characteristics allowing them to be used in internal combustion engines and gas turbines (Oyemakinwa, 2011). Thermochemical conversion process can be demonstrated by the following processes.

#### 2.5.2.1 Pyrolysis

Biomass pyrolysis is a fundamental thermochemical conversion process that is of both industrial and ecological importance. Pyrolysis in itself is used to convert biomass into liquid fuel or bio-oil. It can be divided into three subclasses which are slow pyrolysis, fast pyrolysis and flash pyrolysis (Ruiz et al., 2013). Pyrolysis of solid state materials, such as biomass, can be classified as a heterogeneous chemical reaction. The reaction dynamics and chemical kinetics of heterogeneous processes can be affected by three key elements which are the breakage and redistribution of chemical bonds, changing reaction geometry and the

interfacial diffusion of reactants and products. Unlike homogeneous reactions, concentration is a inconsequential parameter that cannot be used to monitor the progress of heterogeneous reaction kinetics because it can vary spatially. Heterogeneous reactions usually involve a superposition of several elementary processes such as nucleation, adsorption desorption, interfacial reaction and surface or bulk diffusion, each of which may become rate-limiting depending on the experimental conditions (White et al., 2011).

Amongst the thermochemical processes, pyrolysis has received increasing attention since the process conditions may be optimised to produce high energy density pyrolytic oils in addition to the derived char and gas (Kucuk & Dermirbas, 1995). For example, thermal degradation of cellulose proceeds through two types of reaction: a gradual degradation, decomposition, and charring on heating at lower temperatures, and a rapid volatilization accompanied by the formation of levoglucosan on pyrolysis at higher temperatures. The rates of these reactions and the course of the overall decomposition process are also highly dependent on the ambient atmosphere. The initial degradation reactions include depolymerization, hydrolysis, oxidation, dehydration, and decarboxylation. The nature and extent of many individual reactions involved in this process are not known or are insufficiently defined. However, it is known that these reactions are highly influenced by: the temperature and period of heating; the ambient atmosphere, which affects the mass and heat transfer, particularly the supply of oxygen, water, and other reacting or inert gases; and the composition and physical nature of the substrate, especially with respect to inorganic impurities and additives (Kucuk & Dermirbas, 1995).

#### 2.5.2.2 Gasification

Gasification is a thermochemical partial oxidation process in which carbonaceous substances (biomass, coal and plastics) are converted into gas in the presence of a gasifying agent (air, steam, oxygen, carbon dioxide or a mixture of these), The gas generated, commonly referred to as syngas (synthesis gas), consists mainly of hydrogen, CO, CO<sub>2</sub>, N<sub>2</sub>, small particles of char (solid carbonaceous residue), ashes, tars and oils (Ruiz et al., 2013). The versatility of gasification is that it can be used for producing syngas, H<sub>2</sub> or other liquid fuels and can thereby meet the demand for electricity or thermal energy. Furthermore, the resulting fuel can be transported with high energy densities, enabling the generation of electricity to be centralized based on the disperse gasification system (Ruiz et al., 2013).
Biomass gasification has yet to become consolidated as a mature technology, and in most markets it cannot compete with other methods of energy conversion. According to Dasappa, the performance of a 100 kW gasification plant with a downdraft reactor (a type of reactor in which biomass and the gasifying agent come into contact via parallel flows) connected to the grid is unsatisfactory, operating over 1000 h and supplying energy to the grid for only 70 h. The key issues to be faced when designing a gasification plant are the gasifier, its operation and the treatment and adaptation of the syngas generated, without forgetting the paramount importance of biomass preparation and logistics. These two latter aspects are not the primary focus of this paper, but this should not be taken to mean they are any less important (Ruiz et al., 2013).

Gasification may appear to be a rigid technology, as it requires a thorough adaptation of the fuel to be processed and, once this has been achieved and the operating parameters have been fine- tuned, it allows little operating flexibility. This means that any variations in the specific characteristics of the biomass will have unwanted consequences for the gasification process, such as operating instability, loss of performance, problems of scaling, etc. needed, preferably prior to entry in the gasifier. For gasification, moisture content should be between 10% and 15% (Ruiz et al., 2013).

### 2.6 Torrefaction Process

Torrefaction is a low temperature thermal conversion that improves the fuel properties of biomass. It is also known as the mild form of pyrolysis where the torrefaction process occurs at atmospheric pressure and the temperature ranging of 200-300°C with the absence of oxygen and presence of nitrogen (Boateng & Mullen, 2012). The inert or nitrogen condition provides a hydrophobic condition for the biomass, due to the removal of the hydroxyl group during thermal treatment. Therefore, the torrefied biomass provides suitable chemical and physical characteristics for long-distance transportation and long term storage (Na et al., 2013). The primary goal of torrefaction process is to refine biomass to an upgraded solid fuel, including better handling qualities and enhanced combustible properties simile to fossil coal's, leading to de-creased costs, but financial gains. The essential principle in this respect is to increase the energy density of the biomass, requiring a growth of the ratio between energy and mass. Consequently the calorific value of the torrefied biomass increases as well, since it is a specific value reflecting the released energy per mass unit for solid fuels (Schorr et al., 2012). During the torrefaction process the input biomass loses about 30 % of its mass,

but only 10 % of its energy, due to the degassing of low-energy volatile compounds and the escape of moisture, eventuating in a higher energy density of the biomass of roughly 30 % more energy per mass unit. However, there are even more advantages of the torrefied biomass, when compared to the untreated feedstock biomass or conventional wood pellets (Schorr et al., 2012). The grindability of the input biomass can be increased significantly by torrefaction due to the modification of its molecular structure, so that existing problems arising with untreated biomass in the milling component of a coal power plant are overcome. Also the biomass exchanges its hydrophilic properties to hydro-phobicity that allows an effortless storage that goes hand in hand with a greater resistance against biological degradation, self-ignition and physical decomposition in general. However, the risk of biological degradation is not overcome completely, but fungal growth and microbial activity is reduced, since the torrefied material stays very dry. Since the torrefied product already loses a great amount of volatiles during the thermo-chemical conversion, there are less remaining for the following combustion step. That might lead, maybe even more than for conventional biomass, to lower emissions in terms of "sulphur dioxide (SO2), nitrogen oxides (NOx) and net greenhouse gas emissions of carbon dioxide (CO2)", but also a diminished level of ash formation, respectively a new composition of the inorganic residues. As one can expect, each of these issues has a tremendous potential in both ecological and economical views (Schorr et al., 2012).

Torrefaction is a technology that was developed from the coffee industry, where the coffee beans get roasted to make them brittle and to gain their special flavors for the final product. For the biomass industry, the first pilot plant of torrefied biomass was engineered and built in France by the company of Pechiney in the mid of 1980 (Schorr et al., 2012). It was served on the purpose of a reducing agent in an aluminium production process but not for energy reasons. In a global view now, there is one location where R&D referring to torrefaction is focused which is the Netherlands, together with Belgium, where several demonstration plants are either being planned or already operated in the test of procedures (Schorr et al., 2012).

The advantages of torrefaction are significant in the way of improving the physical characteristics of biomass and thus the overall economics of the biomass utilization process for energy production. Torrefied biomass is a homogenous solid fuel with higher energy content and lower moisture content (Dutta & Leon, 2011). As mentioned earlier, torrefied biomass is hydrophobic and this enables it to be negligible towards biological activities such as decomposition and mold. Besides that it also makes the pelletization process to be carried

out easily. However, there are still some disadvantages of torrefaction process. Despite higher calorific value after the process, the energy density is not improved significantly. Although the size is smaller, some of the energy content in original biomass is lost. Until today, there is still limited knowledge on process performance, properties of torrefied product and composition on volatiles from the process of torrefaction (Dutta & Leon, 2011).

#### 2.7 Summary

Basically, this chapter was mainly discussed on the recent literature review of this study. It included the review of biomass and its types, palm oil industry in Malaysia and the raw materials used. Besides that, the study of different biomass conversion techniques and methods also have been done in this chapters.

# **CHAPTER 3**

# **MATERIALS AND METHODS**

### 3.1 Chapter Overview

This chapter will present the material and methodology that need to apply for the study. It will have a general description on the list of material that needed to use for the experimental study. Besides that, the methodology to run the experiment will be stated as well in this chapter.

### 3.2 Biomass Sample

The palm oil solid wastes needed in this study are empty fruit bunches (EFB), mesocarp fiber and kernel shell. All of these solid wastes are obtained from the LKPP Lepar Palm Oil Mill located in Gambang, Pahang. For the experiment purpose, the solid wastes will be broken down into fibrous form and powder form as one of the parameter needed. The fibrous form of solid waste is produced by using the grinder and a pair of scissors in order to cut it into a uniform length. While the powder form of solid waste is produced by using a blender bought for the experimental purpose. Figure 3.1 and 3.2 showed the fibrous and powder form of particles obtained for the experiment.



Figure 3-1: Fibrous form of I.) mesocarp fiber, II.) EFB and III.) kernel shell.



Figure 3-2: Powder form of I.) mesocarp fiber, II.) EFB and III.) kernel shell.

### 3.3 Gases

There is only one gases involves in the whole study of torrefaction process. Nitrogen gas will be used to ensure the elimination of oxygen from the pyrolyzer along the process taking place. However, there is only a small portion of nitrogen (0.5 L/min) needed along the experiment.

### 3.4 Characterization of Moisture Content

The characterization of moisture content for each of the types of solid wastes either in the form of fibrous or powder is determined by using the electric oven which is located in the Environmental Engineering Lab of University Malaysia Pahang (UMP). The samples are prepared and placed into the crucibles. The crucibles are then inserted into the electric oven for duration of an hour at the temperature of 105 °C. The initial weight and the final weight of the samples are recorded and calculation is done to get the value of moisture content. The apparatus used is shown at Figure 3.3 below.



Figure 3-3: Electric Oven in Environmental Engineering Lab UMP

#### 3.5 Characterization of Calorific Value

The calorific value of each sample is determined by using the bomb calorimeter which was located at the Basic Engineering Lab of UMP. The steps of determining the calorific value was followed exactly the same experiment of bomb calorimeter carried out in the course of Basic Engineering Lab.

The experiment started in with the step a combustion capsule was being cleaned and dried, meanwhile, 1 g of sample was weighed accurately by using the analytical capsule. The sample was then filled into the combustion capsule and followed by fixed it on the bomb head. 10 cm of fuse wire was cut and the wire was attached on the bomb head by rising up the cap, followed by the wire inserted through the eyelet, a "U" shape was made and the cap was pulled downward. The fuse wire was ensured to immerse or touch the sample but should not touch the combustion capsule to prevent short circuit. This was followed by the attached of bomb head with them combustion bomb until it is tight. The bomb was filled with oxygen gas while the oval bucket was filled with 2 L of distilled water accurately. The lifting handle was attached to the two holes in the side of the screw cap and the combustion bomb was lowered into the water. The combustion bomb was handled carefully so the sample will not be disturbed. It was necessary to ensure there was no leaking of bubbles coming out from the combustion bomb. The handle was removed and any drops of water are shaking back into the bucket. The ignition lead wire was pushed into the terminal sockets on the bomb. The cover was put vertically on the jacket with the thermometer facing toward the front. The stirrer was turned on in order to ensure that it runs freely. The drive belt was slipped onto the pulleys and the motor was started. The stirrer was run and the temperature was recorded at one minute intervals. After one minute, the bomb was fired by pressing and holding the ignition button until the indicator lights goes out. The temperature was read and recorded at one minute intervals until the temperature being constant for at least 3 reading.

After the last temperature reading, the motor was stopped, the belt was removed and the cover was lifted from the calorimeter vertically. The cover was put on the support stand. The ignition leads wire was removed and the bomb was lifted out of the bucket. The bomb was wiped with a clean towel. The knurled knob on the bomb was opened and head slowly to release the gas pressure. After all pressure has been released (no sound), the cap was unscrewed and the head was lifted out of the cylinder and was placed on the support stand. All the unburned pieces of fuse wire were removed from the bomb electrodes, being

straightened and their combined length was measured in centimetre. The calorific value can be obtained from the measurement tape according to their length. The steps were repeated for all the samples either in fibrous or powder form. Figure 3.4 below showed the structure of the bomb calorimeter.



Figure 3-4: Structure of Bomb Calorimeter

#### **3.6 Characterization of FTIR Test**

FTIR stands for Fourier Transform Infrared, which was the preferred method of infrared spectroscopy. In infrared spectroscopy, IR radiation was passed through a sample and some of the infrared radiation was absorbed by the sample and some of it was passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. Just like fingerprint, no two unique molecular structures produced the same infrared spectrum. This makes infrared spectroscopy useful for several types of analysis. In this study, FTIR was used to identify the component and chemical compositions which exist on the surface structure of the samples before and after torrefaction process. The analysis has been done by using the Thermo Scientific FT-IR Spectrometer located at the Faculty of Industrial Science and Technology of University Malaysia Pahang.

#### **3.7 Torrefaction Process**

The process was carried out by using a tubular type reactor that was made by University Malaysia Pahang. The experiment was carried out in two parts, first was the study on the effect of the total surface area of biomass particles (fibrous form and powder form) towards the process and followed by the effect of different temperature on the calorific value, mass yield and the energy yield. The experiment was started by setting all the parameters needed in the experiment at the control system of the pyrolyzer. A total of three patterns were set at the temperature of 473K, 523K and 573K. Then the experiment started with an amount of biomass sample in fibrous form was weighed and put on a crucible. The crucible was then inserted into the tube and placed on the center of reactor. Nitrogen gas was then inserted to the reactor and flushed for 15 minutes in order to eliminate the oxygen inside. This was followed by the step of the selecting of pattern needed which was the temperature at 473K at first with a constant rate of 25°C/min by an electric furnace surrounding the reactor. The heater was switched off after 30 minutes of torrefaction process and the reactor was left to cool down to an ambient temperature. The torrefied sample was collected, weighed and keep in an air tight vessel till the characterization. The steps above were repeated by replacing the fibrous form of biomass to the powder form of biomass. Each experiment was repeated for at least three times (Uemura et al., 2011). Figure 3.5 and Figure 3.6 below showed the control system and the main structures of the pyrolyzer used.



Figure 3-5: Control system of Pyrolyzer used



Figure 3-6: Main structure of Pyrolyzer used

#### **3.8 Measurements**

The moisture content and the calorific value were measured before and after the experiment for all the biomass samples used in this study. The moisture content of the sample was measured by using an electric oven. The sample was placed at a crucible before it placed into the oven for an hour at the temperature of 105°C. The initial and final weights of samples are recorded. The moisture content was calculated based on the equation below.

$$MC = \frac{Initial \ weight - Final \ Weight}{Initial \ weight} \times 100\%$$

The calorific value was measured using a bomb calorimeter in the basic chemistry lab of University Malaysia Pahang and the CV value was determined based on the length of unburned wire collected. The calorific value from a bomb calorimeter was the high heat value (HHV), which included the latent heat of the vapour emitted from the specimen. While the other three parameters were calculated by the equations below,

$$Ym = \frac{Mass \ of \ solid \ after \ torrefaction}{Mass \ of \ EFB \ used}$$
$$CV \ ratio = \frac{CV \ of \ solid \ after \ torrefaction}{CV \ of \ EFB \ used}$$

$$Ye = Ym \times CV \ ratio$$

Where Ym means the mass yield, CV means the calorific value and Ye means the energy yield (Uemura et al., 2011).

# **CHAPTER 4**

# **RESULTS AND DISCUSSION**

### 4.1 Chapter Overview

This chapter consist of result and discussion for the research. First, the preliminary results on the moisture content and the calorific value before the experiment was tabulated. Next is followed by the calibration curve on the effects of temperature towards the calorific value for all the samples. This is then followed by the discussion on the mass and energy yields for all the three samples in this study. The last part of this chapter is discussed on the FTIR analysis before and after the torrefaction process for all the samples.

### 4.2 Preliminary Results on Moisture Content and Calorific Value

Types of Sample	Fibrous	Form(g)	Moisture Content (%)	Powder	Form(g)	Moisture Content (%)
	Before	After		Before	After	
Fiber	1	0.935	6.5	1	0.950	5.0
	1	0.915	8.5	1	0.930	7.0
	1	0.925	7.5	1	0.955	4.5
		Average	7.5		Average	5.5
EFB	1	0.790	21.0	1	0.950	5.0
	1	0.825	17.5	1	0.900	10.0
	1	0.805	19.5	1	0.910	9.0
		Average	19.3		Average	8.0
Kernel Shell	1	0.880	12.0	1	0.900	10.0
	1	0.850	15.0	1	0.930	7.0
	1	0.875	12.5	1	0.940	6.0
		Average	13.2		Average	7.7

Table 4-1: Value of Moisture Content for each of the sample

Type of Sample	Fibrous Form (cal/cm)	CV(MJ/kg)	Powder Form (cal/cm)	CV(MJ/kg)
Fiber	6.0	25.104	8.0	33.472
	4.0	16.736	9.0	37.656
	5.5	23.012	7.0	29.288
	Average $= 5.2$	21.617	Average $= 8.0$	33.472
EFB	5.0	20.920	5.0	20.920
	6.0	25.104	7.0	29.288
	4.0	16.736	6.0	25.104
	Average $= 5.0$	20.920	Average $= 6.0$	25.104
Kernel Shell	4.0	16.736	7.5	31.380
	5.0	20.920	10.0	41.840
	8.0	33.472	9.0	37.656
	Average $= 5.7$	23.709	Average $= 8.8$	36.959

Table 4-2: Value of CV for each of the sample before torrefaction process

Table 4.1 and 4.2 displayed the results of torrefacation process at various temperatures which are 473K, 523K and 573K respectively. Table 4.1 and table 4.2 showed the result of moisture content and calorific value of samples before the torrefaction process. For both the characterization of moisture content and calorific value, 1`g of sample was used to test on the value. As can be seen in table 4.1, after the calculation, the average value of moisture content in percentage for the mesocarp fiber, EFB and kernel shell in the fibrous form were 7.5, 19.3 and 13.2 respectively. While in the form of powder, the respective moisture content in percentage for mesocarp fiber, EFB and kernel shell were 5.5, 8.0 and 7.7. While for the calorific value, the average values obtained for the fibrous form sample of mesocarp fiber, EFB and kernel shell are 21.62 MJ/kg, 20.92 MJ/kg and 23.71 MJ/kg respectively. The average values of powder form sample of mesocarp fiber, EFB and kernel shell were 33.47 MJ/kg, 25.10 MJ/kg and 36.96 MJ/kg respectively. The torrefaction results will further discuss according to the effects of temperature on the calorific value and the effects of temperature on the mass yield and energy yield by each sample in only fibrous form. This is because the powder form of samples has fully decomposed after the torrefaction process which resulted at a point where no mass yield is collected and hence, no energy yields from the samples. Below are the figures of sample collected after torrefaction process for all the samples.



Figure 4-1: Samples of EFB after torrefaction



Figure 4-2: Samples of mesocarp fiber after torrefaction



Figure 4-3: Samples of kernel shell after torrefaction

# 4.3 Effects of temperature on the calorific value



Figure 4-4: Effects of temperature on CV

Figure 4.4 showed the trend of CV of different samples at various temperatures of 473K, 523K and 573K. It is obviously showed that the CV has increased accordingly with the increased in temperature for all the samples of EFB, mesocarp fiber and kernel shell. For the case of EFB, the average CV before torrefaction process was 21.62 J/g, and is increased to 30.47 J/g at 473K, 32.08 J/g at 523K and 33.68 J/g at 573K respectively which showed an increment of 40-55% at different temperatures. While for mesocarp fiber, the average CV has increased from an initial value of 21.62 J/g to a value of 34.64 J/g at 473K, 35.29 J/g at 523K and lastly 36.89 J/g at 573K which showed an increment of 63-70% at the three temperatures. For the last sample which was the kernel shell, the average CV is increased from an initial value of 23.71 J/g to 33.68 J/g at 473K, 35.29 J/g at 523K and lastly 35.93 at 573K which showed an increment of 42-52% at the three temperatures tested.

Similar to the result obtained from Uemura et al., (2011), the trend of CV which is the HHV obtained from bomb calorimeter increased significantly after torrefaction process at various temperatures. Similarly in this study, the trend of CV increased gradually for all the samples after the torrefaction process. All the three types of samples showed a steady increase at the temperature of 473K to 573K as figure 4.1 indicated a straight line graph for all the three samples. Among all the samples, EFB has the lowest CV at all the temperatures compared to mesocarp fiber and kernel shell. Mesocarp fiber has the highest CV of 36.89 J/g at the temperature of 573K which is also the highest CV compared to EFB and kernel shell after torrefaction process. Therefore, the sequence of the highest CV started from the mesocarp fiber, followed by the kernel shell and lastly the EFB.



4.4 Effects of temperature of the mass yield and energy yield

Figure 4-5: Effects of temperature on the average mass and energy yield (EFB)



Figure 4-6: Effects of temperature on the average mass and energy yield (mesocarp fiber)



Figure 4-7: Effects of temperature on the average mass and energy yield (kernel shell)

Figures 4.5, 4.6 and 4.7 showed the results of the effects of temperature on mass and energy yield after the torrefaction process respectively. It can be obviously seen that the average mass yield of each samples decreased gradually after the samples being torrefied. The average mass yield by EFB indicated the most significant lost after torrefaction which were 48.67% at 473K, followed by 37% at 523K and lastly 29.67% at 573K. While for the case of mesocarp fiber, the average mass yields were 67% at 473K, 55% at 523K and 51% at 573K. Lastly for the case of kernel shell, the average mass yields were 82.67% at 473K, followed by 73.33% at 523K and lastly 66% at 573K.

As the torrefaction temperature increases, the mass yield decreased steadily, whereas the decreasing ratio is depend on the waste type; as can be seen, EFB has the highest decreasing ratio and kernel shell has the lowest. This trend of study is consistent with the study of Uemura, et al., 2011 where its trend of study was the same as current study. The reduction in mass yield was mainly due to the two major reasons which were the losing of moisture content and the thermal decomposition of structure to form volatile or gaseous products such as  $H_2O$ , CO,  $CO_2$  and so on. The decreased in mass for the temperature of 473K which was the lowest operating temperature of torrefaction process was mainly due to the losing of moisture such as 523K and 573K was attributed to the thermal decomposition of the hemicellulose part of the biomass. Comparison between EFB, mesocarp fiber and kernel shell showed an obvious result, which EFB has the lowest value among the three samples which means that

EFB decomposed more than the other two during the torrefaction process. This was then followed by mesocarp fiber and lastly the kernel shell.

For the study of energy yield, the range of the samples yield was from 48.53% to 109.59% depending on the mass yield of the samples at different temperature. Again, EFB has the lowest energy yield while mesocarp fiber has the highest energy yield among the three samples. The average energy yield for EFB was 58.05% at 473K, 57.88% at 523K and 48.53% at 573K. While for mesocarp fiber, the average energy yield was 88.12% at 473K, 90.44% at 523K and 86.88% at 573K. Lastly for the case of kernel shell, the average energy yield was 95.86% at 473K, 109.59% at 523K and 100.2% at 573K. According to Uemura et al., 2011, the energy yield of the dried samples would be 100% if only moisture loss during the treatment. In this study, all the samples showed a trend of moisture loss except for the kernel shell at temperature of 523K and 573K with the value of 109.59% and 100.2% respectively. Both the trend of EFB and mesocarp fiber showed a condition of moisture loss at all the values of temperature tested with the values of energy yield of less than 100%. This can be explained that the samples have decomposed to some extent during the torrefaction process. This is indirectly contributed to the total mass yield of all the samples where the average mass yield of each samples are also the factors that affected the result of lower energy yield by the samples. This can be seen clearly as the trend of decreased in mass yield has also affected the trend of energy yield to be decrease for all the samples.

### 4.5 FTIR Analysis



Figure 4-8: FTIR analysis for EFB, I.) 473K, II.) 523K, III.) 573K



Figure 4-9: FTIR analysis for mesocarp fiber, I.) 473K, II.) 523K, III.) 573K



Figure 4-10: FTIR analysis for kernel shell, I.) 473K, II.) 523K, III.) 573K

			EFB			Fiber			Shell	
Wavelengt		473	523	573	473	523	573	473	523	573
$h (cm^{-1})$	Bonds	Κ	Κ	Κ	Κ	Κ	Κ	Κ	Κ	Κ
3700-3000	O-H	А	А	А	А	А				А
3000-2800	C-H	А	А	А	А	А	А	А	А	А
1800-1650	C=O				А			А	А	
1650-1500	C=C	А	А	А		А	А			А
1450-1200	C-H	А	А	А					А	А
1200-950	C-O	А	А					А	А	А
900-750	C-H									

Table 4-3: Bonds present before and after torrrefaction process (A stands for Available)

Table 4.3 summarized the bonds present after the torrefaction process at three different temperatures from the results obtained from Figure 4.8, 4.9 and 4.10. As can be seen from Table 4.3 above, the bonds present before, the bond presents before the torrefaction process were the OH bonds, CH bonds, C=O bonds, C=C bonds, and CO bonds. OH bonds were detected at the region of 3700-3000 cm<sup>-1</sup> while for the case of CH bonds; it can be detected in three regions which were 3000-2800 cm<sup>-1</sup>, 1450-1200 cm<sup>-1</sup> and 900-700 cm<sup>-1</sup>, for both aliphatic and aromatic compounds. The double bond structures in were in range of 1800-1650 cm<sup>-1</sup> and 1650-1500 cm<sup>-1</sup> for C=O and C=C bonds respectively. Both of these compounds showed the present of cellulose and lignin in the structure. Lastly was the aliphatic alcohol of CO bonds which were detected at the region of 1200-950 cm<sup>-1</sup>.

It was assumed that the structure of materials was the same for all the three components before the torrefaction process as oil palm solid biomass was mainly made up by cellulose, hemicellulose and lignin. From the results obtained, it can be seen that CH bonds at 900-750 cm<sup>-1</sup> were diminished for all the samples after the torrefaction process. For EFB, the chemical bonds remained almost unchanged except the breakdown of C=O bonds at all the temperatures and CO bonds at 573 K. While for the mesocarp fiber, the chemical bonds that diminished at all the temperatures after the process included the CH bonds of 1450-1200 cm<sup>-1</sup> and the CO bonds. Besides that, the C=O bonds at 523K and 573K and OH bonds at 573K were also breakdown after the process. Lastly for the case of kernel shell, OH bonds of 1450-1200 cm<sup>-1</sup> at 573K and 473K respectively were breaking down after the process. As been discussed, it can be concluded that the structure mesocarp fiber being affected the most while the structure of EFB remained almost unchanged after the process.

### **CHAPTER 5**

### **CONCLUSION AND RECOMMENDATION**

#### 5.1 Conclusion

In this study, torrefaction of empty fruit bunches (EFB), mesocarp fiber and kernel shell which are typically the agriculture solid waste of oil palm biomass in Malaysia, was experimentally conducted. The effect of torrefaction temperature and form of solid biomass on the calorific value, mass and energy yields was investigated for those three types of biomass waste.

As in result for the powder form of samples, it was determined that the samples were fully decomposed at the respective temperature with no result in mass yield, and hence, no results collected for both calorific value and energy yield by the samples. While for the study of fibrous form of samples, according to the study of Uemura et al., (2011), mesocarp fiber and kernel shell showed excellent energy yields values of nearly 100% while EFB exhibited a poor yield of 56%. This study experienced a same trend of results where both kernel shell and mesocarp fiber showed a good energy yield of nearly 100% as well while EFB experienced a poor yield of around 58%. The energy yield was affected by both the calorific value and mass yield of the samples as it was the product of mass yield and CV ratio. By considering the effect of temperature, the calorific value showed a positive trend of increasing with the increase in temperature of process. However, the mass yield decreased steadily with an increase in temperature which has also affected the energy yield of the samples. The results of FTIR analysis showed an almost unchanged of structure in the samples after the process. The diminished of certain bonds was due to the thermal decomposition along the process. In a nutshell, it can be concluded that the use of torrefied biomass as feedstock for further energy application can be done by taking the solid oil palm biomass as raw materials. Optimization need to be done in setting the desired temperature in order to maximize the energy yield of the product.

#### 5.2 Recommendations

The results obtained can be further optimized by ensuring the desired condition of torrefaction process. The following recommendations should be considered in order to ensure an efficient energy application of torrefied biomass. Firstly, the scale of study is in laboratory form of tubular reactor where the sample used was relatively smaller compared to the study of Uemura et al., (2011). Besides that, the samples used for the study was not fresh enough as proper pre-treatment of waste materials was not be done accordingly. This was mainly due to the reason of the collected solid wastes was left to expose instead of treated it with proper drying condition. While for the case of bomb calorimeter, the handling procedures need to be done accordingly in order to obtain a high precision of results. Precautions such as avoiding the fuse wire from touching the surface of combustion capsule in the bomb calorimeter should be taken seriously from time to time during the experiment. A touching of the fuse wire with the surface of combustion capsule will results in a short circuit which causes the fuse wire to burn before the explosion. Hence, this will directly affect the length of fuse wire collected in the experiment where the remaining fuses wire is used to determine the CV of the sample.

During the torrefaction process, the first thing that needs to be ensured is the condition inside the tubular reactor. The flushing of nitrogen is compulsory to confirm that the process was carried on under the absence of oxygen. Besides that, the handling of samples need to be done carefully where the position of samples put should be at the center of the reactor in order to ensure a proper heating process. Cooling down of reactor a need to be taking care before proceed to the next torrefaction process in order to obtain a better result.

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# **APPENDICES**

Temperature (K)	Sample	Unburn wire (cal/cm)	CV value (J/g)	CV ratio (%)	Mass Yield (%)	Energy Yield (%)
473	1	6.900	28.870	138.000	48.000	23.040
473	2	10.350	43.304	207.000	53.000	109.710
473	3	4.600	19.246	92.000	45.000	41.400
Average		7.283	30.473	145.667	48.667	58.050
523	1	11.500	48.116	230.000	40.000	92.000
523	2	5.750	24.058	115.000	36.000	41.400
523	3	5.750	24.058	115.000	35.000	40.250
Average		7.667	32.077	153.333	37.000	57.883
573	1	11.500	48.116	230.000	32.000	73.600
573	2	6.900	28.870	138.000	28.000	38.640
573	3	5.750	24.058	115.000	29.000	33.350
Average		8.050	33.681	161.000	29.667	48.530

Table 1: CV of EFB after torrefaction process

Table 2: CV of mesocarp fiber after torrefaction process

Temperature (V)	Sampla	Unburn wire	CV value	CV ratio	Mass Yield	Energy Yield
(K)	Sample	(cal/cm)	(J/g)	(%)	(%)	(%)
473	1	8.050	33.681	155.809	68.000	46.240
473	2	6.900	28.870	133.550	63.000	84.137
473	3	9.890	41.380	191.422	70.000	133.996
Average		8.280	34.644	160.261	67.000	88.124
523	1	9.660	40.417	186.971	60.000	112.182
523	2	8.050	33.681	155.809	55.000	85.695
523	3	7.590	31.757	146.905	50.000	73.453
Average		8.433	35.285	163.228	55.000	90.443
573	1	9.200	38.493	178.067	50.000	89.034
573	2	8.050	33.681	155.809	53.000	82.579
573	3	9.200	38.493	178.067	50.000	89.034
Average		8.817	36.889	170.648	51.000	86.882

Temperature (K)	Sample	Unburn wire (cal/cm)	CV value (J/g)	CV ratio (%)	Mass Yield (%)	Energy Yield (%)
473	1	9.200	38.493	162.355	85.000	72.250
473	2	8.050	33.681	142.061	83.000	117.910
473	3	6.900	28.870	121.766	80.000	97.413
Average		8.050	33.681	142.061	82.667	95.858
523	1	6.900	28.870	121.766	70.000	85.236
523	2	9.200	38.493	162.355	75.000	121.766
523	3	9.200	38.493	162.355	75.000	121.766
Average		8.433	35.285	148.826	73.333	109.590
573	1	9.660	40.417	170.473	68.000	115.922
573	2	8.050	33.681	142.061	65.000	92.340
573	3	8.050	33.681	142.061	65.000	92.340
Average		8.587	35.927	151.532	66.000	100.200

Table 3: CV of kernel shell after torrefaction process

Following tables were the raw data of the characterization of calorific value after torrefaction,

# EFB at 200°C,

	Sample 1	Sample 2	Sample 3
Mass of sample (gram)	0.50	0.50	0.50
Unburn fuse wire (cal/cm)	6.90	10.35	4.60

Time (minute)	Т	Cemperature reading (°C	C)
Time (minute)	Sample 1	Sample 2	Sample 3
1	26.5	27.0	27.5
2	26.7	27.2	27.8
3	26.8	27.4	28.0
4	27.0	27.5	28.1
5	27.0	27.6	28.1
6	27.0	27.6	28.1
7		27.6	

EFB at 250°C,

	Sample 1	Sample 2	Sample 3
Mass of sample (gram)	0.50	0.50	0.50
Unburn fuse wire (cal/cm)	11.50	5.75	5.75

Time (minute)	Г	Cemperature reading (°C	C)
Time (initiate)	Sample 1	Sample 2	Sample 3
1	27.0	27.5	27.8
2	27.2	27.8	28.0
3	27.4	28.0	28.2
4	27.5	28.1	28.3
5	27.6	28.1	28.3
6	27.6	28.1	28.3
7	27.6		

# EFB at 300°C,

	Sample 1	Sample 2	Sample 3
Mass of sample (gram)	0.50	0.50	0.50
Unburn fuse wire (cal/cm)	11.50	6.90	5.75

Time (minute)	Т	<sup>°</sup> Cemperature reading ( <sup>°</sup> C	C)
	Sample 1	Sample 2	Sample 3
1	27.5	28.0	28.3
2	27.8	28.2	28.5
3	28.0	28.5	28.6
4	28.3	28.6	28.7
5	28.3	28.6	28.8
6	28.3	28.6	28.8
7			28.8

# Fiber at 200°C,

	Sample 1	Sample 2	Sample 3
Mass of sample (gram)	0.50	0.50	0.50
Unburn fuse wire (cal/cm)	8.05	6.90	9.89

Time (minute)	Г	Cemperature reading (°C	C)
	Sample 1	Sample 2	Sample 3
1	28.0	28.3	28.5
2	28.2	28.5	28.8
3	28.5	28.7	29.0
4	28.6	28.8	29.1
5	28.7	28.8	29.2
6	28.7	28.8	29.2
7	28.7		29.2

# Fiber at 250°C,

	Sample 1	Sample 2	Sample 3
Mass of sample (gram)	0.50	0.50	0.50
Unburn fuse wire (cal/cm)	9.66	8.05	7.59

Time (minute)	Т	Cemperature reading (°C	C)
Time (minute)	Sample 1	Sample 2	Sample 3
1	27.5	28.0	28.3
2	27.8	28.2	28.5
3	28.0	28.5	28.6
4	28.3	28.6	28.7
5	28.4	28.6	28.7
6	28.4	28.6	28.7
7	28.4		

# Fiber at 300°C,

	Sample 1	Sample 2	Sample 3
Mass of sample (gram)	0.50	0.50	0.50
Unburn fuse wire (cal/cm)	9.20	8.05	9.20

Time (minute)	Г	Cemperature reading (°C	2)
Time (minute)	Sample 1	Sample 2	Sample 3
1	28.8	29.0	29.4
2	29.0	29.2	29.6
3	29.1	29.3	29.8
4	29.2	29.6	29.9
5	29.3	29.6	30.1
6	29.3	29.6	30.1
7	29.3		30.1

Shell	at	<b>200</b> °	°C,
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	Sample 1	Sample 2	Sample 3
Mass of sample (gram)	0.50	0.50	0.50
Unburn fuse wire (cal/cm)	9.2	8.05	6.9

Time (minute)	Т	Cemperature reading (°C	C)
Time (initiate)	Sample 1	Sample 2	Sample 3
1	26.5	27.0	27.5
2	26.7	27.2	27.8
3	26.8	27.4	28.0
4	27.1	27.5	28.1
5	27.1	27.7	28.3
6	27.1	27.7	28.3
7		27.7	28.3

# Shell at 250°C,

	Sample 1	Sample 2	Sample 3
Mass of sample (gram)	0.50	0.50	0.50
Unburn fuse wire (cal/cm)	6.90	9.20	9.20

Time (minute)	Г	Cemperature reading (°C	2)
Time (minute)	Sample 1	Sample 2	Sample 3
1	27.8	28.0	28.3
2	27.9	28.2	28.5
3	28.0	28.5	28.6
4	28.3	28.7	28.8
5	28.5	28.7	29.0
6	28.5	28.7	29.0
7	28.5		29.0

# Shell at 300°C,

	Sample 1	Sample 2	Sample 3
Mass of sample (gram)	0.50	0.50	0.50
Unburn fuse wire (cal/cm)	9.66	8.05	8.05

Time (minute)	Temperature reading (°C)		
	Sample 1	Sample 2	Sample 3
1	28.8	29.1	29.5
2	29.0	29.3	29.7
3	29.1	29.5	29.9
4	29.2	29.7	30.1
5	29.5	29.7	30.2
6	29.5	29.7	30.2
7	29.5		30.2