

Effect of Atmosphere and Biomass Form on torrefaction

by

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Dissertation submitted in partial fulfilment of
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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Chemical Engineering Programme
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(CHEMICAL ENGINEERING)

Approved by,

(Prof. Dr. Yoshimitsu Uemura)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

NOOR AZIAH BINTI OTHMAN

ABSTRACT

Torrefaction of palm oil empty fruit bunches (EFB), mesocarp fibre and kernel shell, wastes from the palm oil industry, was carried out in a fixed bed tubular reactor in the presence of oxygen at different concentrations from 0 to 15 % (nitrogen balance). The effects of torrefaction conditions, oxygen concentration (0, 3, 9 and 15 %), temperature (493, 523 and 573 K) and biomass size (0.375, 1.5, 3 and 6 mm), on the mass and energy yields were investigated. The mass yield decreased with an increase in temperature and oxygen concentration, but was not affected by biomass size. The energy yield decreased with an increase in oxygen concentration. It was found that oxidation was occurring along with torrefaction in this project. The ultimate analysis was also conducted.

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Nomenclature and Abbreviation

N ₂	Nitrogen
O ₂	Oxygen
CO ₂	Carbon Dioxide
EFB	Empty Fruit Bunch
C	Carbon element
H	Hydrogen element
N	Nitrogen element
S	Sulphur element
TGA	Thermogravimetry Analysis
LHV	Lower Heating Value
HHV	High Heating Value
CV	Calorific Value

CHAPTER 1

1.0 INTRODUCTION

1.1 Background of Project

Energy consumption is relatively proportional to the development. International Energy Agency (IEA) in 2009 World Energy Outlook projected that between 2007 and 2030, world primary energy demand will increase by 1.5% per year [1]. Primary energy is subjected to natural resources including coal, crude oil, and natural gas. These resources contain high percentage of carbon but they cannot be regenerated in a short time. The dead organisms take millions of year to decompose into the fuels. Therefore, alternatives must be introduced in order to cope with the vigorous growth of energy demand.

Biomass is a very promising alternative for this problem since it is largely abundant. In Malaysia, more than 70 million tonnes per year of biomass is produced mainly from palm oil industry [2]. However, the energy density of biomass is lower (15-19GJ/tonne) compared to coal (20-30GJ/tonne). Higher moisture contents might have results in this problem [3]. Therefore, biomass should undergo treatment process so that more significant energy content is obtained.

BIOMASS FEEDSTOCK AVAILABILITY	
Source	Dry Tons/Year
Fuelwood	17,000,000
Urban wood residues	20,000,000
Logging slash & site clearing	41,000,000
Restoration & fuel treatment	15,000,000
Increased forest residual	65,000,000
Annual crop residues	286,000,000
Perennial crops	156,000,000
Agricultural process residues & manure	84,000,000
Total	684,000,000

Figure 1: Biomass feedstock availability. *Source: HM3 Energy, Inc.* retrieved from <http://hm3e.com/torrefied-biomass/BiomassFeedstockAvailability.php> [4]

Several articles have reported the thermal treatment called torrefaction in order to improve fuel properties of biomass [5,6,7,8,9]. Torrefaction is a thermal treatment that occurs in an inert atmospheric condition [5,6]. According to [5,7,8], torrefaction is carried out within temperature range of 200 to 300°C.

The product of torrefaction is mainly in a form of solid usually called as torrefied biomass. As per year 2008, J. Poldervaart, MD Polow Energy Systems by, has noted several processes that are practiced in torrefaction industry (refer Table 1).

Table 1: Torrefaction in Practice [10]

Processes	Suppliers	Advantage/disadvantages
<i>Batch processes</i>	-	Based on production of char; conventional Not to say old fashioned; high cost
<i>Continue processes</i>		
Rotary kilns	Wysmont Stramproy CDS	Known technology, problematic inertisation Long residence times, big dimensions, High investment and maintenance cost
moving bed	ECN a.o.	Big pressure drops in the bed (bed has to be moved), difficult up scaling, only restricted control
Torbed	Polow	Diffusion restriction; very well controllable compact and relatively low in investment low in maintenance

Torrefaction process is known to improve the fuel properties of biomass such as increase in fixed carbon and ash content, and decrease in moisture content and volatile matter [5], [6]. In this report, detail discussion related to these properties will be discussed.

1.2 Problem Statement

Torrefaction process should be done under inert atmosphere with no presence of O_2 or CO_2 . This is because; biomass oxidation will occur if oxidants exist. The oxidation causes loss of chemical energy in the biomass, in other words, less calorific value. In previous report, the torrefaction gas in used is Nitrogen (N_2) gas. N_2 can give good quality of fuel.

However, the cost of N_2 gas has contributed the largest portion in total cost for torrefaction process relatively. There is no report found to date that evaluates the cost of N_2 gas specifically. Most of the economic evaluations discussed the capital investment of the project [11].

In order to optimize the torrefaction cost, there is new approach to use flue gas from industry as torrefaction gas rather than N_2 gas. Other than cost free, the temperature of flue gas is higher than ambient temperature which is around $500^\circ C$. Therefore, no additional heating source will be acquired to heat up the process. However, this innovation will have drawback since flue gas contains O_2 and CO_2 gasses. These two gasses will cause oxidation to biomass.

Therefore, in this research work, the author will study the capability of flue gas to be used as torrefaction gas in the process. The allowable concentration of O_2 and CO_2 that will give good quality of torrefied biomasses will be determined by mixing N_2 gas and air before introducing the gasses into the torrefaction reactor. The author will use air as the replacement of CO_2 and O_2 gasses because air contains both gasses and require no cost.

1.3 Objectives and Scope of Study

The purpose of this project is to investigate the following variables on torrefaction of Malaysian lignocellulosic biomass.

- a) The effects of atmosphere which in this case are concentration of O₂ and CO₂ gasses.
- b) The effects of biomass shape and size.

Scope of study for this research project is to perform analysis on the effect of using mixture of N₂ gas and air (contains CO₂ and O₂) and different biomass sizes in torrefaction process. Only the biomass wastes in Malaysia will be utilized in this research work. This study will focus on biomass waste from palm oil mill which include empty fruit bunch (EFB), mesocarp fiber and kernel shell. The analysis of this study will be based on the literature review and will be continue with the experimental work.

CHAPTER 2

2.0 LITERATURE REVIEW

2.1 Lignocellulosic Biomass

Lignocellulose or biomass that originates from plants, generalises the structure of plants to three main structures; cellulose, hemicellulose and lignin. These three polymeric structures are mainly considered in most of the studies to understand the decomposition mechanisms of woody and herbaceous biomass. They form the foundation of cell walls and provide mechanical strength and tenacity (toughness) to plant structures.

A typical plant cell has structure as shown in Figure 2 below. It can be described into primary and secondary walls. The three structures are located in secondary wall. Each cell is connected by middle lamella which functions as glue.

This secondary wall mainly consists of cellulose and is very well organised by nature. The cellulose microfibrils are embedded in a matrix of hemicellulose that bonds the microfibrils mechanically, but also through hydrogen bonding. The cell wall has a repetitive pattern in which hemicellulose binds microfibrils of a cell wall and lignin binds adjacent cells. The function of hemicellulose is often well illustrated by comparing its function to concrete in reinforced concrete. Without the concrete the iron rods lose their mutual coherence and orientation [12].

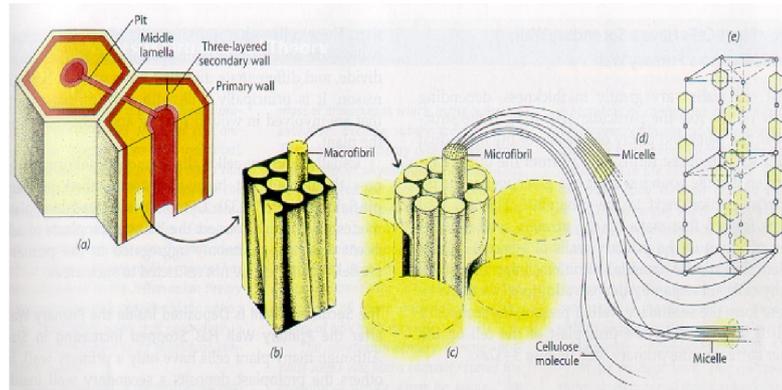


Figure 2: Detailed impression of the structure of a cell wall. (a) Part of the cell wall and middle lamella, primary wall and secondary cell wall, (b) macrofibril mutual structure, (c) microfibril structure, (d) individual cellulose polymers including micelles, and (e) mutual coherence of individual cellulose polymers on a micro level (entrained flow gasification)

2.2 Properties of Biomass Fuel

Demirbas. A, (2002) has discussed the difference between fuel properties of biomass and coal. The fuel density of coal is 61% higher than biomass which is very significant. Meanwhile, the particle size of coal is much finer than biomass. This parameter may also influence the heating value. Nevertheless, author does not find any report specifically discussed the effect of particle size on fuel properties. In Table 2, it shows that carbon content of coal is the highest among red oak wood and wheat straw. This support the fact that coal has higher dry heating value than biomass because the dry heating value is largely contributed by fixed carbon content.

Table 2: Physical Properties and heating Values of Biomass and Coal Fuels [13]

Property	Biomass	Coal
Fuel density (kg/m ³)	~500	~1300
Particle size	~3 mm	~100 μm
Dry heating value (MJ/kg)	16	25

	C	H	N	S	Cl	Ash	O (diff.)
Coal	81.5	4.0	1.2	3.0	–	7.0	3.3
Red oak wood	50.0	6.0	0.3	–	–	1.3	42.4
Wheat straw	41.8	5.5	0.7	–	1.5	15.0	35.5

Figure 3: Ultimate Analyses of Typical fuel Samples [13]

2.3 Torrefaction

2.3.1 Research Work

Many research works were being carried out related to torrefaction. There are reports found regarding the effects of torrefaction on fuel qualities and combustion [5], torrefaction of wood, weight loss kinetics and grindability [14,15,8], and also techno – economic evaluation [11]. The common parameters that are evaluated for the torrefied biomass are calorific value and ultimate analysis [5,7,8,16]. Biomasses that have been studied included willow, beech, larch, straw, reed canary grass birch, pine and bagasse [12,5,16]. The author found no paper discussed the torrefaction of palm oil waste.

The torrefaction experiment was carried out in a small scale (5-10 g sample) fixed bed torrefaction reactor [7,14]. Some papers also reported torrefaction

together with TGA measurements carried out with a Perkin-Elmer Pyris 6 TG apparatus with auto sampler. This enables the observation of weight loss during torrefaction [5,15].

2.3.2 Decomposition Mechanism during Torrefaction

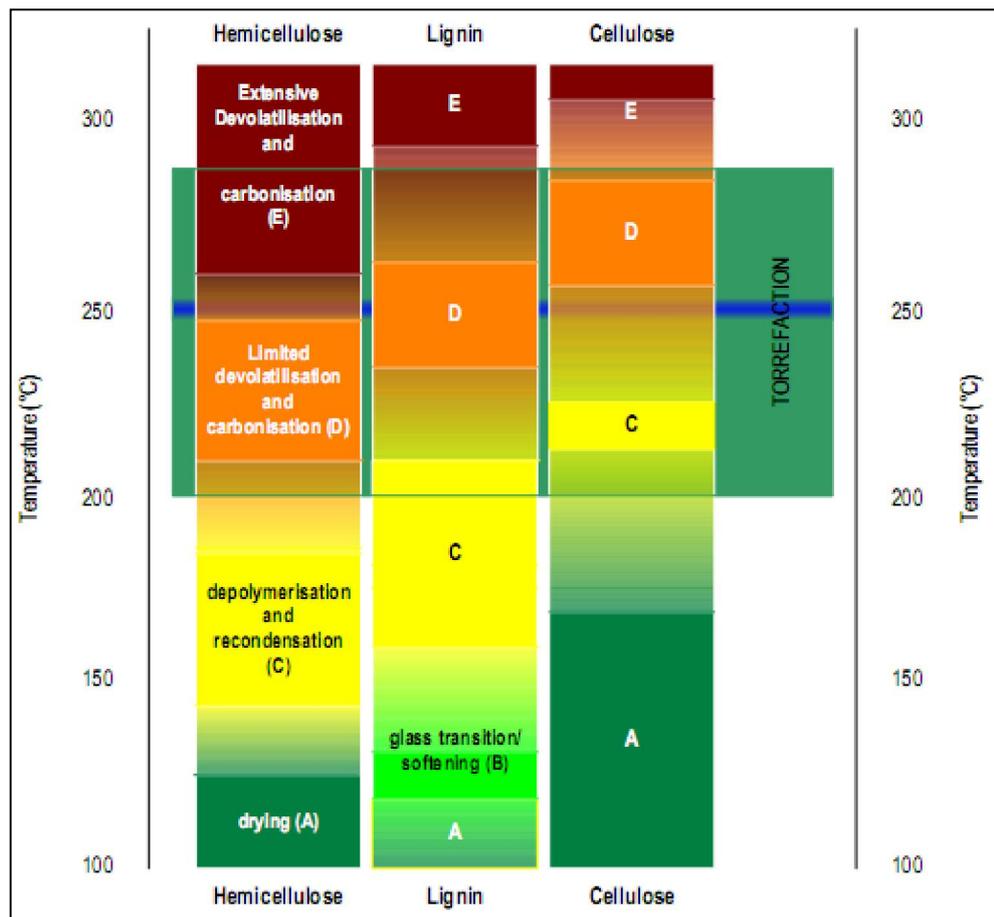


Figure 4: Main decomposition regimes of lignocelluloses during torrefaction [6].

Based on the figure taken from [6], in temperature regime A, physical drying of biomass occurs. When the temperature is increased to regime C, depolymerisation occurs and the shortened polymers condense within the solid structure. In regime D, limited devolatilisation and carbonisation of the intact polymers and the solid structures formed in the temperature regimes C. Further increase of temperature to regime E leads to extensive devolatilisation and carbonisation of the polymers and the solid products that were formed in regime D. For lignin, it undergoes a temperature regime B which softening of it occurs.

The torrefaction temperature regime and the blue line splits the regime into a low (<250 °C) and high temperature regime (>250 °C). In general hemicellulose is the most reactive polymer followed by lignin and cellulose is most thermostable. This shows that hemicelluloses decompose at lower temperature.

2.3.3 Torrefaction Time and Temperature

According to [12], before torrefaction time is introduced, the term residence time was used. However, it only expresses the hold-up time of biomass in a torrefaction reactor. It does not tell how long actual torrefaction takes place, since part of the residence time is 'lost' due to heating of the biomass possibly in combination with drying. Figure 5 shows the time-temperature characteristics of several stages during torrefaction process. When moist biomass of ambient temperature is fed into a batch torrefaction reactor, the biomass is first heated to a temperature at which the biomass is dried. Then the temperature further increases until the desired torrefaction temperature is

reached (200°C to 300°C). This temperature is maintained until the reactor is cooled again.

Only in this temperature range the torrefaction decomposition reactions occur. This range can be described by three time-temperature phases. First the biomass is heated from 200°C to the desired torrefaction temperature (T_{tor}) in period $t_{\text{tor,h}}$. Then the temperature is hold for period t_{tor} at the torrefaction temperature, until cooling during period $t_{\text{tor,c}}$. The decomposition reactions will occur mainly during t_{tor} , but this will depend on the time contribution of the heating and cooling period. The reaction time has been defined as the sum of $t_{\text{tor,h}}$ + t_{tor} and thus leaving out the cooling time $t_{\text{tor,c}}$.

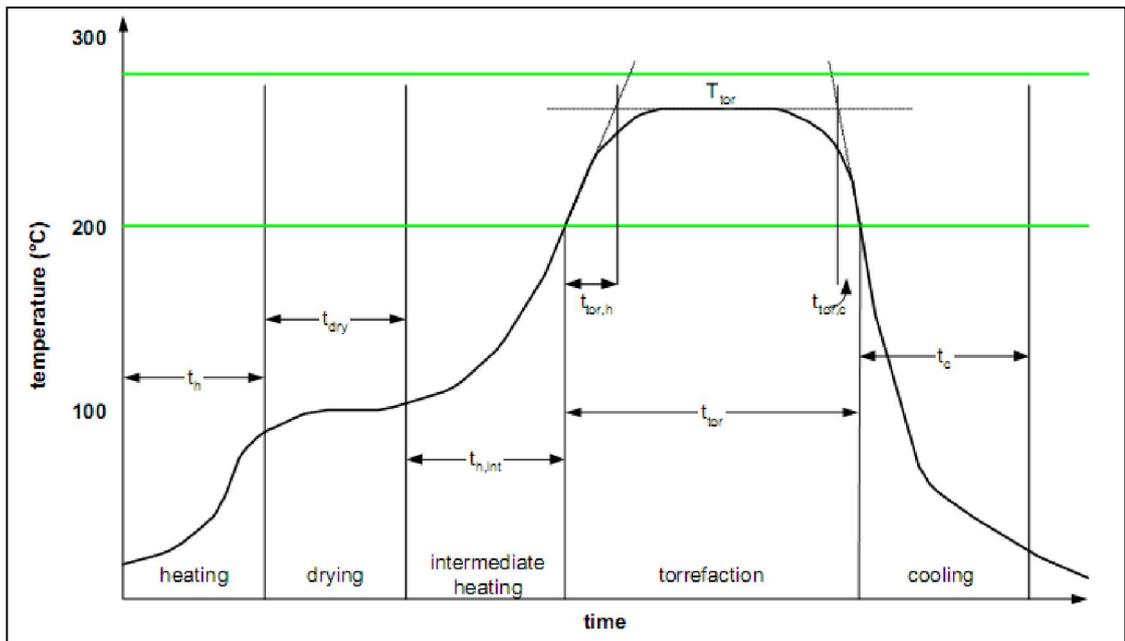


Figure 5: Stages in the heating of moist biomass from ‘ambient’ temperature to the desired torrefaction temperature and the subsequent cooling of the torrefied product [12].

2.4 Torrefied Biomass

2.4.1 Mass and energy yield

The mass and energy yield are main parameters in the evaluation of the torrefaction process. Based on [5], mass and energy yield can be defined as equation (1) and (2) respectively

$$Y_{mass} = 100\% \times (\text{mass after drying or torrefaction} / \text{mass of wet sample before the treatment}) \quad (1)$$

$$Y_{ene} = Y_{mass} \times (LHV \text{ after treatment} / LHV \text{ before treatment}) \quad (2)$$

Mass yield is influenced by torrefaction temperature as shown in Figure 6, where the mass loss of biomass is parallel with final torrefaction temperature. The mass yield of the biomass is also depending on the composition of the biomass. Biomass that contains more hemicelluloses will experience more mass loss than the other biomass that contains less hemicellulose. This is because, hemicelluloses decompose at lower temperature. Therefore, at the final torrefaction temperature, hemicelluloses are mostly decomposed that results in great mass loss. This theory is proved by the relation of Figure 7 and Figure 8 [5]. Wheat straw which contains the highest hemicelluloses (30.8%), resulted in the lowest mass yield (55.1%) at temperature 563K. However, the energy yield by wheat straw is significant at this temperature despite the mass loss that is 65.8% (Figure 9). This shows that torrefaction improves energy yield of the biomass.

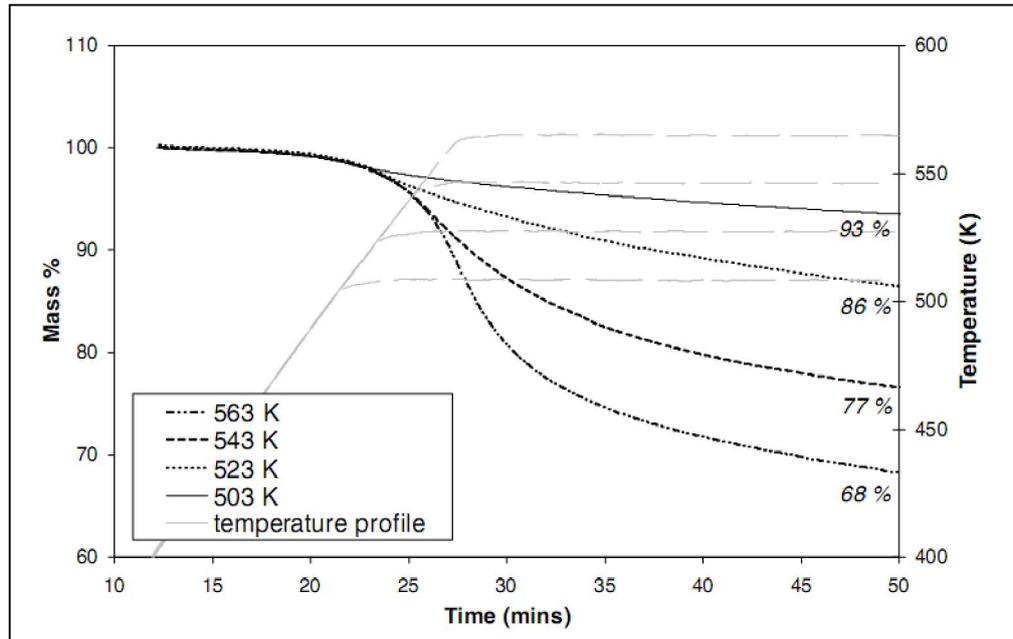


Figure 6: Mass loss during the torrefaction of reed canary grass at different final temperatures [5].

	Lignin (%)	Cellulose (%)	Hemicellulose (%)	Total (DMD) (%)
Reed canary gass	7.6	42.6	29.7	80.0
Wheat straw	7.7	41.3	30.8	79.8
Willow	20.0	49.3	14.1	83.4

Figure 7: Mass% of hemicelluloses, cellulose and lignin in raw biomass fuels [5].

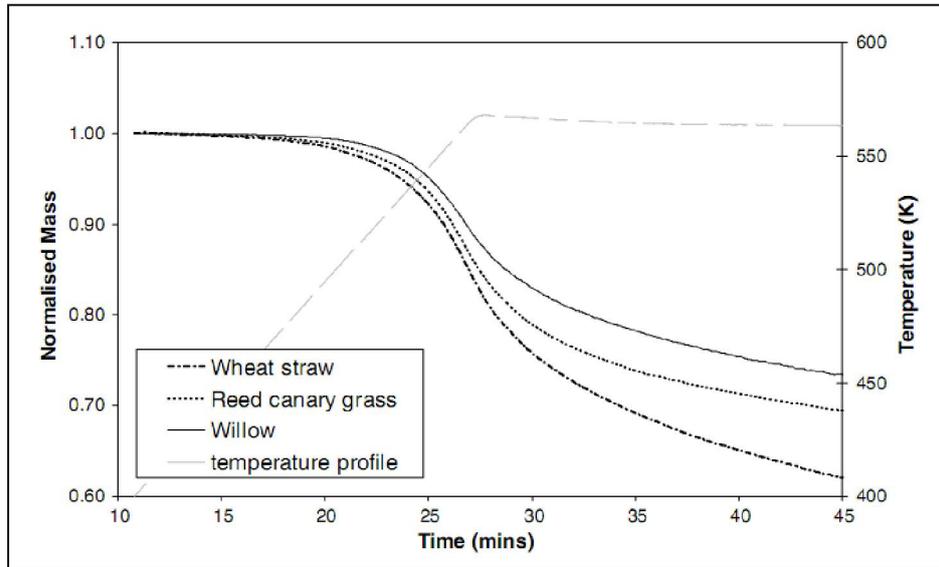


Figure 8: Mass loss of wheat straw, reed canary grass and willow during torrefaction at 563 K [5].

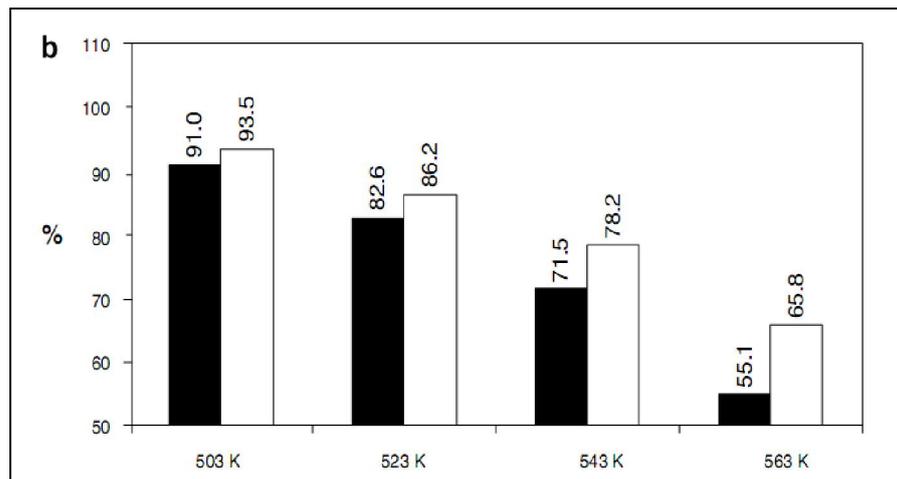


Figure 9: Mass and energy yields (dry ash free) for wheat straw [5].

2.4.2 Ultimate analysis, calorific value and moisture content

	Raw	Torrefaction temperature (K)			
		503	523	543	563
<i>RCG</i>					
C (%)	48.6	49.3	50.3	52.2	54.3
H (%)	6.8	6.5	6.3	6.0	6.1
N (%)	0.3	0.1	0.0	0.1	0.1
O (%)	37.3		37.0	37.3	36.3
Moisture (%)	4.7	2.5	1.9	1.3	1.2
CV (kJ/kg)	19,500	–	20,000	20,800	21,800

Figure 10: Ultimate analysis, calorific value, and moisture content of untreated and torrefied reed canary grass [5].

Based on the above figure retrieved from [5], fixed carbon content of the reed canary grass increases when the torrefaction temperature increases. Other than that, the calorific value of the biomass also increases with the torrefaction temperature. Prins relates both fixed carbon content and calorific value are by equation (3). From the equation, the value of coefficient **a**, **b**, **c**, and **d** are decreasing respectively. Therefore, as **a** is the coefficient for fixed carbon content, **C**, the value of **C** give the greatest influence to calorific value.

$$\text{HHV [MJ/kg]} = a \cdot C + b \cdot H + c \cdot O + d \quad (3)$$

Meanwhile, the moisture content is decreased when the torrefaction temperature increase.

2.4.3 Ash content

Ash, the material remaining, calculated on the basis of the dry weight of the original sample, after the sample is ignited at a specified temperature. The ash content of the sample may consist of: (1) various residues from chemicals used in its manufacture, (2) metallic matter from piping and machinery, (3) mineral matter in the pulp from which the paper was made, and (4) filling, coating, pigmenting and/or other added materials. The amount and composition of the ash is a function of the presence or absence of any of these materials or others singly or in combination [20]. Torrefaction also increase the ash content of the fuel. Biomass torrefied at higher temperature results in higher in ash content [5, 9]. Up to date, there is no specific explanation found to justified this statement. Ash is a solid, particulate, inorganic combustion residue. Of forest fuels, ash content varies between different components, stem wood 0, 4-0, and 6%, and stem bark 2-5% and 1-2% branches. The ash content is highest in those parts of the tree where growth occurs. Ash from the wood fuel contains nutrients which the tree raised, including important trace elements. Nitrogen (N) is missing because it largely leaves in gaseous combustion. Since trees take up heavy metals and radioactive substances from soil and air, are also those substances in the ash. Generally, the ash is between 10% and 30% of calcium (Ca). The content of potassium (K) and magnesium (Mg) is usually a few percent, while the phosphorus (P) represents approximately one percent of the total content [21].

2.5 Flue Gas

Flue gas is gas that exits to the atmosphere via a flue, which is a pipe or channel for conveying exhaust gases from a fireplace, oven, furnace, boiler or steam generator. Quite often, it refers to the combustion exhaust gas produced at power plants. Its composition depends on what is being burned, but it will usually consist of mostly nitrogen (typically more than two-thirds) derived from the combustion air, carbon dioxide (CO₂) and water vapor as well as excess oxygen (also derived from the combustion air). It further contains a small percentage of pollutants such as particulate matter, carbon monoxide, nitrogen oxides and sulfur oxides [17]. Table 3 shows composition of flue gas produced from different combusted material.

Table 3: Composition of flue gas produced according to combusted material [18].

Fuel type	Bituminous coal	Sub-bituminous coal	Natural gas	Natural gas	Fuel oil
Gas (wt)	Utility Boilers			Gas Turbine	Diesel
CO ₂ (%)	18.1	24.0	13.1	5.7	6.2
O ₂ (%)	6.6	7.0	7.6	15.9	17.0
N ₂ (%)	71.9	68.1	79.3	78.4	76.7
SO ₂ (ppm)	3504.0	929.7	0.0	0.0	113.1
NO (ppm)	328.5	174.3	95.1	22.1	169.7
NO ₂ (ppm)	125.9	66.8	36.5	8.5	65.0

Generally, the temperature of flue gas is around 500°C. In Malaysia, palm oil mill is one of the flue gas sources. The flue gas is obtained from steam boiler that is available for the steaming process of fresh fruit bunch. Based on author review, there is project conducted on utilization of flue gas in utilization of flue gas for cultivation of microalgae [19]. No report has been produced about utilization of flue gas as torrefaction gas.

CHAPTER 3

3.0 METHODOLOGY

3.1 Samples

Biomasses from palm oil waste have been tested in the experiment: empty fruit bunch (EFB), mesocarp fibre (MF) and kernel shell (KS). The biomasses were obtained from Felcra Nasaruddin's palm oil mill at Bota, Perak. The biomasses were first dried in drying oven at temperature of 105°C for 24 h. Then, the weight of each biomass was measured every 1 h until the reduction in weight was insignificant. The dried biomasses were grinded and sieved to four size range (Table 4).

Table 4: Different Particle Size of Biomasses

Size Range (mm)	Size Average (mm)
0.25 – 0.50	0.375
1 – 2	1.5
2 – 4	3
4 – 8	6

3.2 Chemicals

Table 5: Chemicals Used in the Studies

Chemical	Purity	Supplier's Name
Purified Nitrogen Gas	99.98 %	MOX - Linde Sdn. Bhd.
Silica Gel	-	Bendosen

3.3 Equipment Set-up

The torrefaction process was carried out in a tubular reactor with diameter of 46mm. The reactor was assembled by the writer as in Figure 11.

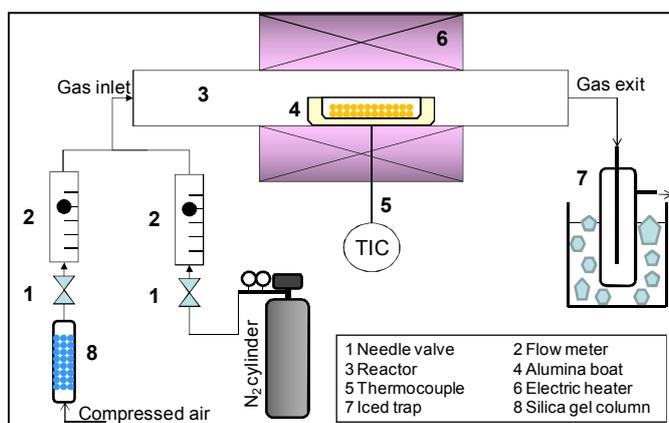


Figure 11: Experimental Apparatus

During the set-up, Methane flowmeter was used to measure air flowrate. Therefore, a calibration curve as shown in Appendix 1 was plotted to calibrate the equipment.

3.4 Experimental Procedure

The grinded sample is filled into the ceramic boat until 1mm full (1-2g) and weighed. Carefully, the ceramic boat is inserted into the reactor by using sample holder (metal wire hand-made holder). Then, the reactor is flushed with torrefaction gas for 15 minutes. After the flushing is completed, the temperature is increased to the desired point (torrefaction temperature) by the rate of 10deg C/min. The torrefaction temperature is maintained for 30 minutes. After 30 minutes, the temperature is set back to 25°C. Throughout the experiment, torrefaction gas with flowrate of 100ml/min is flow through the reactor. The system temperature should be below 30°C before sample can be taken out. The experiment will be repeated by varying four variables that are biomass, temperature, particle size and Oxygen concentration.

Table 6: Study Parameters in the Research

Variables	Variation Level		
Biomass	Empty Fruit Bunch (EFB) Mesocarp Fibre Kernel Shell		
Temperature (°C)	220 250 300		
Biomass Size (mm)	0.375 1.5 3 6		
O ₂ Concentration (%)		Methane Flowmeter (ml/min)	Nitrogen Flowrate (ml/min)
	3	17.5	85
	9	55.5	55
	15	94.0	25

3.5 Analysis

For the biomass wastes (wet) used in this study, the moisture content, calorific value, elementary (CHNS) composition and ash content will be measured. For dried biomass wastes and the torrefied samples, all values except moisture content are measured.

The moisture content will be measured as follows. A prescribed amount of sample (3g) will be weighed in a crucible, and will be placed in an electric oven maintained at 105°C. After 24 h of drying, the sample will be weighed every one hour till the decrease in weight became negligibly small.

The calorific value will be measured using a bomb calorimeter, model C2000 series manufactured by IKA Werke. The calorific value from a bomb calorimeter is the high heat value (HHV), which includes the latent heat of the vapor emitted from the specimen.

Elementary (CHNS) analysis will be carried out using CHNS-932 supplied by LECO Corporation.

The mass and energy yield will be calculate by following equation

$$y_M = \frac{\text{Mass of solid after torrefaction}}{\text{Mass of EFB used}} \quad (1)$$

$$CV \text{ ratio} = \frac{CV \text{ of solid after torrefaction}}{CV \text{ of EFB used}} \quad (2)$$

$$y_E = y_M \times CV \text{ ratio} \quad (3)$$

CHAPTER 4

4.0 RESULTS AND DISCUSSION

Table 1, 2, and 3 shows the EFB, mesocarp fibre, and kernel shell after torrefaction and their physical properties respectively. The calorific value of the untorrefied EFB, mesocarp fibre and kernel shell are 17.43 MJ/kg, 18.60 MJ/kg and 19.89 MJ/kg respectively.

Table 7: Torrefaction results for EFB of 0.375mm

Temp (K)	O2 conc (%)	Calorific Value (MJ/kg)	CV ratio	Mass yield	Energy yield
493	0	17.90	102.66	91.45	93.88
523	0	18.20	104.39	88.14	92.01
573	0	20.83	119.51	79.44	94.94
493	3	17.83	102.28	91.82	93.92
523	3	18.23	104.57	88.71	92.76
573	3	20.85	119.61	79.42	94.99
493	9	17.83	102.29	90.29	92.37
523	9	18.21	104.47	87.40	91.31
573	9	20.84	119.53	76.81	91.81
493	15	17.83	102.28	89.51	91.55
523	15	18.23	104.59	81.38	85.12
573	15	20.83	119.51	72.41	86.53

Table 8: Torrefaction results for Mesocarp Fibre of 0.375mm

Temp [K]	O ₂ conc [%]	Calorific Value [MJ/kg]	CV ratio [%]	Mass yield [%]	Energy yield [%]
493	3	21.25	114.23	94.00	107.37
523	3	21.31	114.59	92.75	106.28
573	3	22.09	118.77	90.34	107.30
493	9	21.05	113.16	93.72	106.06
523	9	21.36	114.83	92.38	106.09
573	9	22.14	119.00	89.80	106.87
493	15	21.10	113.43	93.05	105.55
523	15	21.59	116.07	91.21	105.87
573	15	22.09	118.76	89.53	106.33

Table 9: Torrefaction results for Kernel Shell of 0.375mm

Temp [K]	O ₂ conc [%]	Calorific Value [MJ/kg]	CV ratio [%]	Mass yield [%]	Energy yield [%]
493	3	21.69	109.03	95.80	104.45
523	3	21.97	110.44	94.30	104.14
573	3	22.78	114.53	93.06	106.59
493	9	21.64	108.78	95.44	103.82
523	9	21.92	110.18	93.76	103.31
573	9	22.74	114.30	92.48	105.71
493	15	21.63	108.73	94.88	103.16
523	15	21.85	109.83	93.57	102.77
573	15	22.74	114.28	91.90	105.03

4.1 Effect of Particle Size on Mass Yield

Figures 12 to 20 shows the results of the mass yield of EFB at 493K, 523K and 573K, respectively. From the figures, it is obvious that the mass yield shows no significant dependency on the particle size under the conditions of this study. The same behaviour is also exhibit by mesocarp fibre and kernel shell.

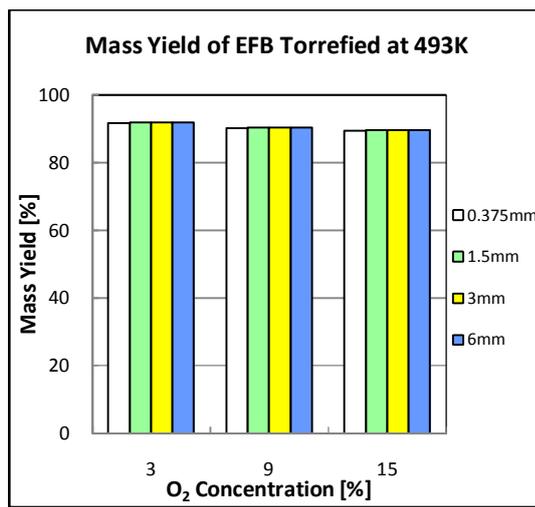


Figure 12: Mass Yield of EFB Torrefied at 493K

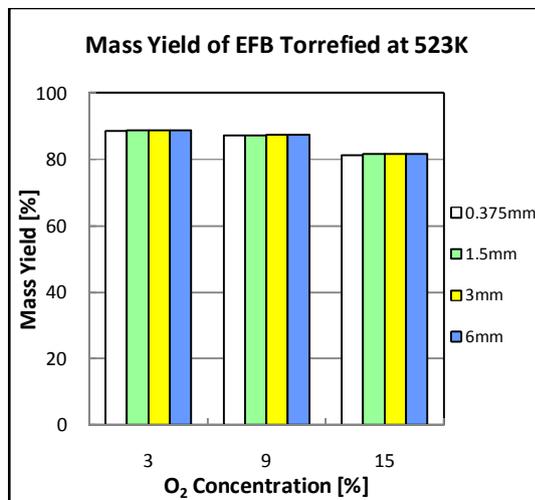


Figure 13: Mass Yield of EFB Torrefied at 523K

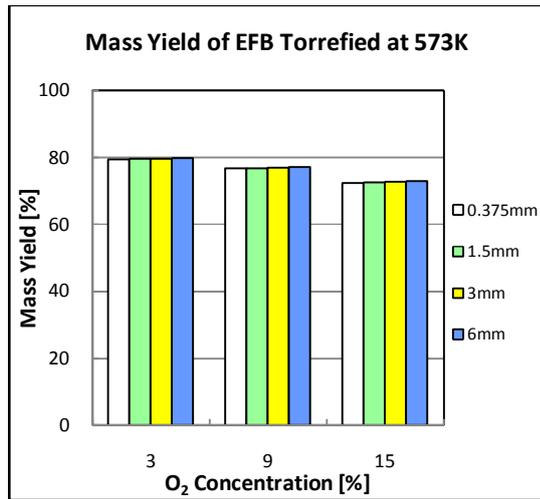


Figure 14: Mass Yield of EFB Torrefied at 573K

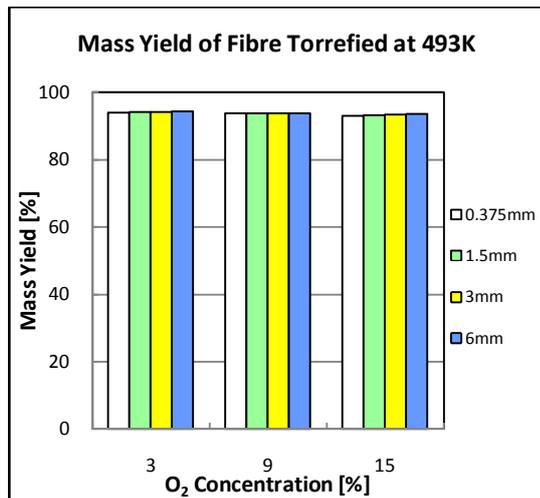


Figure 15: Mass Yield of Fibre Torrefied at 493K

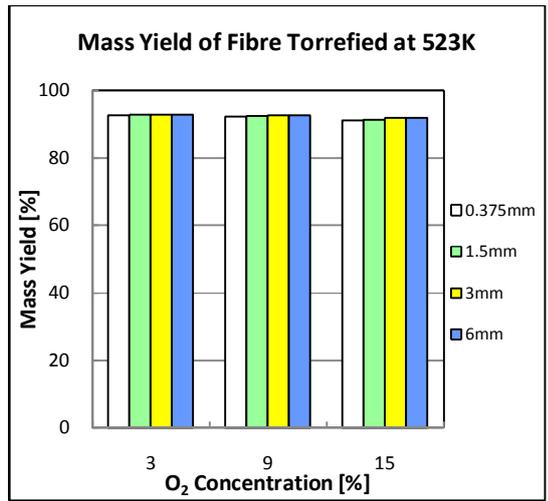


Figure 16: Mass Yield of Fibre Torrefied at 523K

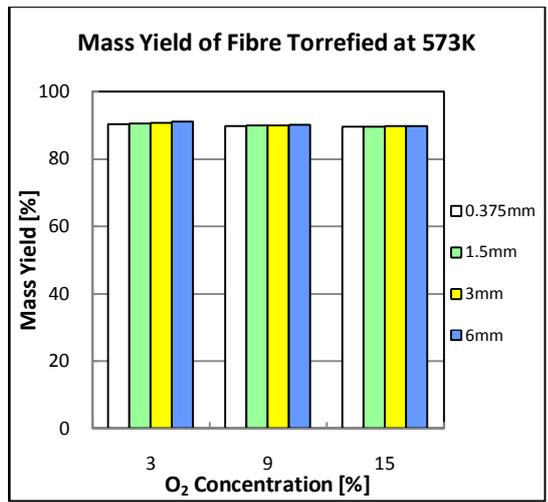


Figure 17: Mass Yield of Fibre Torrefied at 573K

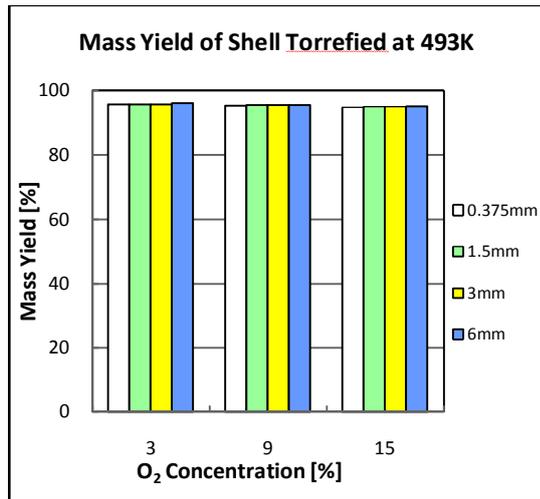


Figure 18: Mass Yield of Shell Torrefied at 493K

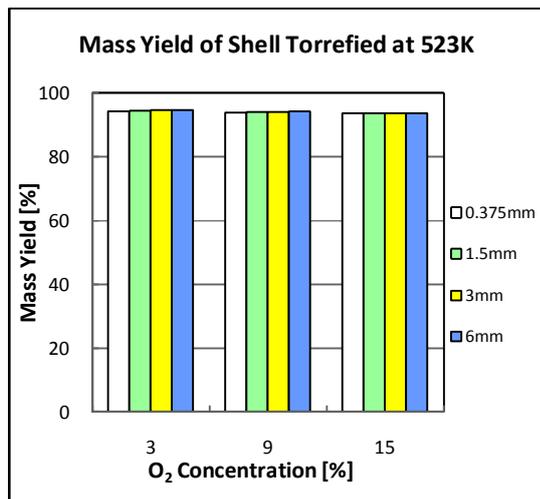


Figure 19: Mass Yield of Shell Torrefied at 523K

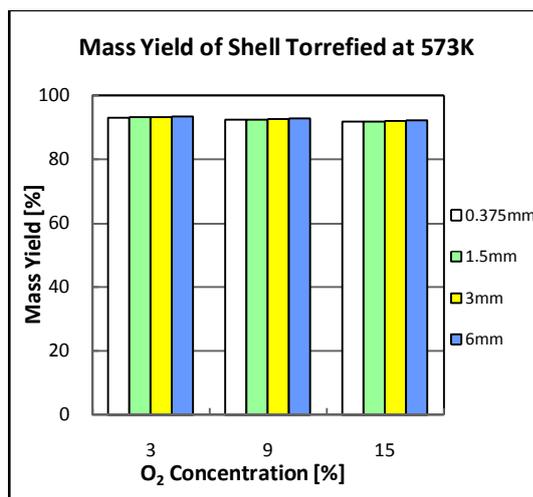


Figure 20: Mass Yield of Shell Torrefied at 573K

4.2 Effect of Temperature and Oxygen Concentration on Mass yield

Figure 21 to 23 show the relationship between mass yield and temperature at different oxygen concentration. In comparison among the three biomasses, EFB shows the lowest mass yield followed by mesocarp fibre and kernel shell. The mass yield of EFB shows dependency to temperature. The mass yield decreases with an increase in temperature. Previous paper [5] reported a similar behaviour, which reflects the positive effect of temperature on the torrefaction rate.

The mass yield also decreases with an increase in oxygen concentration. However, for EFB at 3% oxygen concentration, the mass yield is almost the same as that at 0% oxygen concentration. This result means, in the presence of oxygen, oxidation also occurred concurrently with torrefaction, and is significant in the concentration of more than 3% oxygen in atmosphere.

On the contrary, the mass yield of mesocarp fibre and kernel shell show resistance towards temperature and oxygen concentration. Author suggests that

the microstructure of the biomasses has resulted in such observation. As discussed by T.G. Bridgeman *et. al.*, in the case of torrefaction of reed canary grass, wheat straw and willow, biomass that contains the most hemicelluloses undergoes the most mass reduction. This idea also supported by P. C. A. Bergman *et. al.* who described the mechanism of biomass decomposition during torrefaction. Hemicellulose is the most reactive part that undergoes decomposition compared to celluloses and lignin.

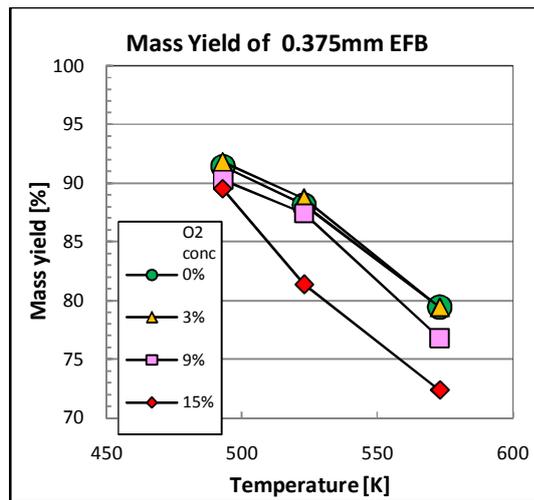


Figure 21: Mass Yield of 0.375mm EFB

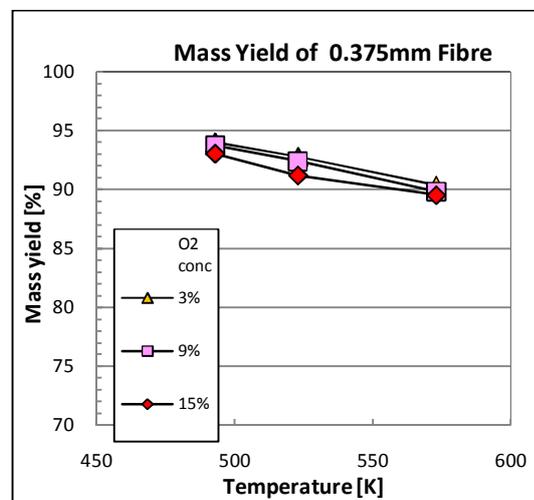


Figure 22: Mass Yield of 0.375mm Fibre

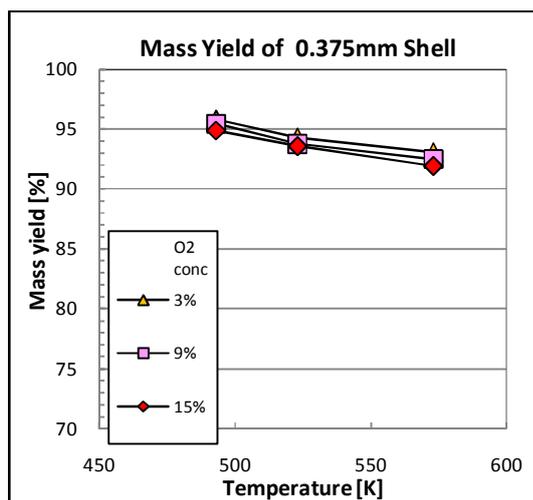


Figure 23: Mass Yield of 0.375mm Shell

4.3 Effect of Particle Size on Calorific Value

Figure 24 to 32 show the relationship between particle size and calorific value. Biomasses with small size give high calorific value. This can be explained by the idea of more components in small particle size EFB has been torrefied compared components in the large particle. Torrefaction is a gas-solid reaction which has low reaction rate. Therefore, 30 minutes residence time which is kept constant in this research might be a major limiting factor to achieve complete torrefaction for 6mm biomasses. As a result, the untorrefied components of biomasses will bring down the calorific value.

However, for each biomass the behaviour is slightly different. For EFB and kernel shell, the effect is not very significant. However, for mesocarp fibre, 0.375mm particle give the highest calorific value while the other are almost the same when torrefied at 493K and 523K (see Figure 27 and 28). Meanwhile, for mesocarp fibre torrefied at 573K (see Figure 29), particles of 0.375 mm and 1.5mm show clear increment of the calorific value. Most likely, the behaviour exhibit by each biomass is depending on their nature microstructure.

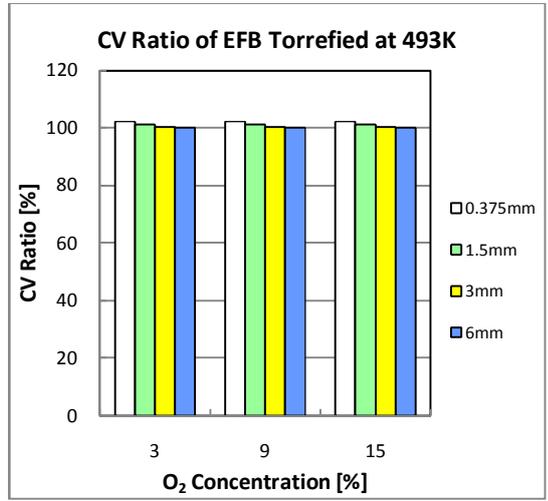


Figure 24: CV Ratio of EFB Torrefied at 493K

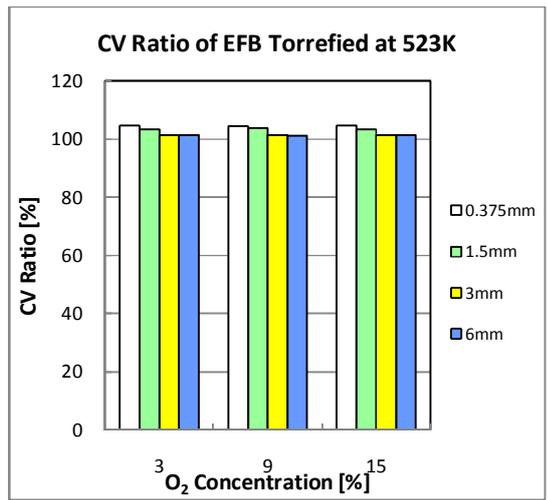


Figure 25: CV Ratio of EFB Torrefied at 523K

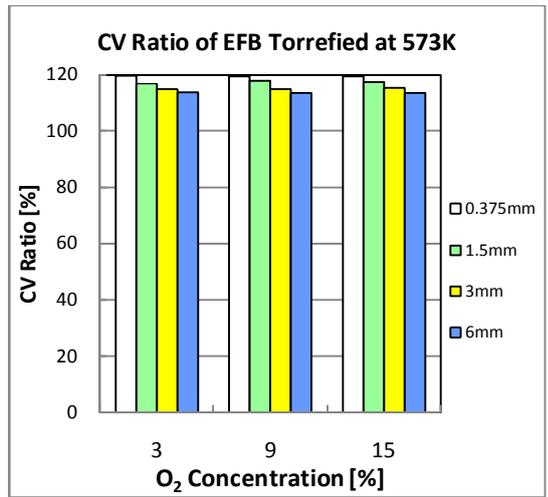


Figure 26: CV Ratio of EFB Torrefied at 573K

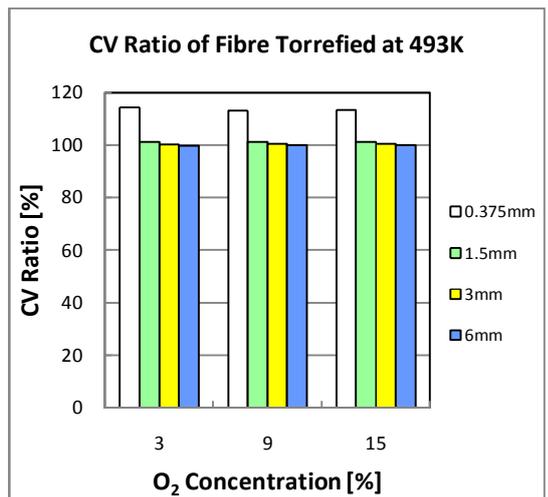


Figure 27: CV Ratio of Fibre Torrefied at 493K

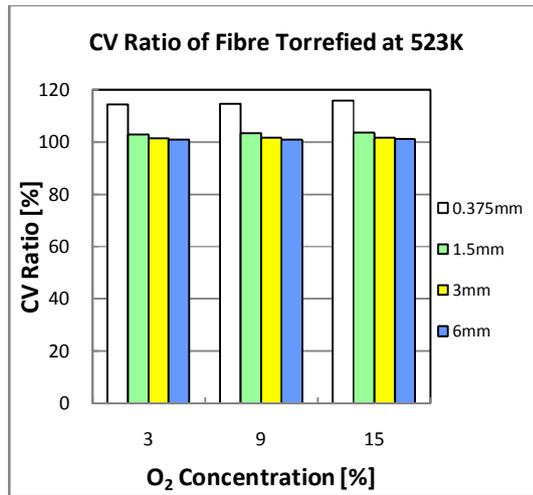


Figure 28: CV Ratio of Fibre Torrefied at 523K

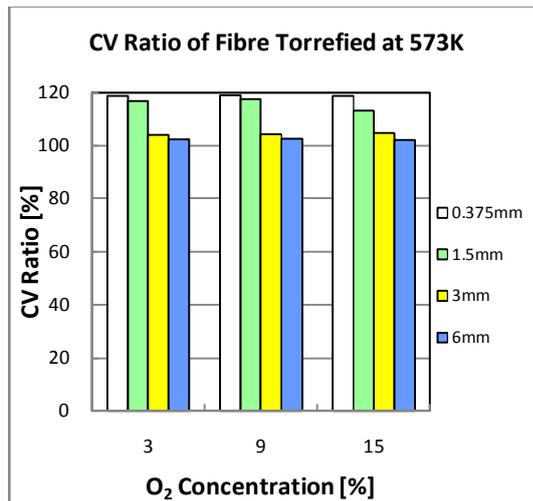


Figure 29: CV Ratio of Fibre Torrefied at 573K

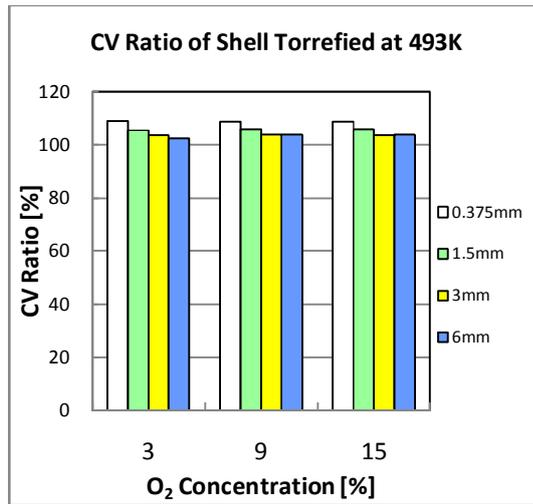


Figure 30: CV Ratio of Shell Torrefied at 493K

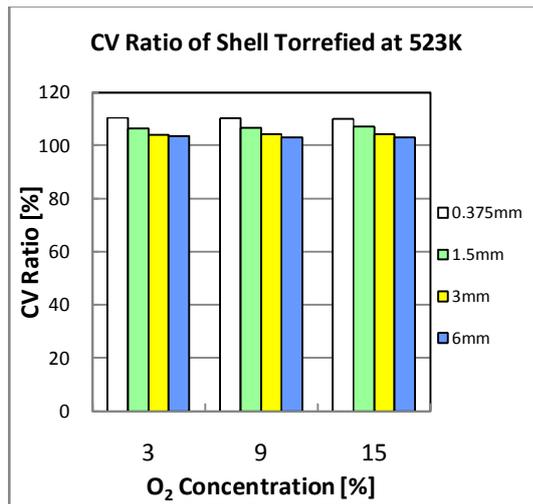


Figure 31: CV Ratio of Shell Torrefied at 523K

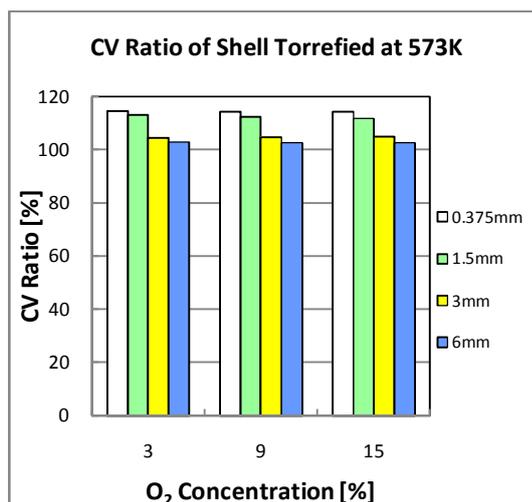


Figure 32: CV Ratio of Shell Torrefied at 573K

4.4 Effect of Temperature and Oxygen Concentration on Calorific Value

Figure 33 to 35 show the relationship between calorific value and temperature at different oxygen concentration. The calorific value increases with an increase in temperature. Prins and colleague also reported the same tendency, in which wood and grass-type lignocellulosic biomass samples were used. It can be explained by the fact that the main gaseous products during torrefaction are water and carbon dioxide [14]. However, the calorific value has no dependency on oxygen concentration in the range of 0 to 15% (see Figure 33). The authors cannot find any report, in which torrefaction is conducted in the existence of oxygen.

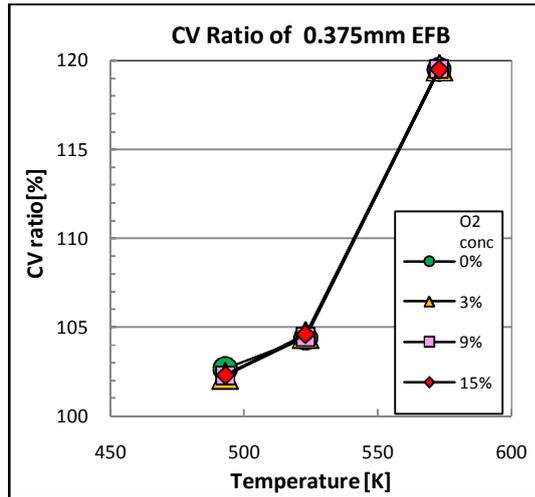


Figure 33: CV Ratio of 0.375mm EFB

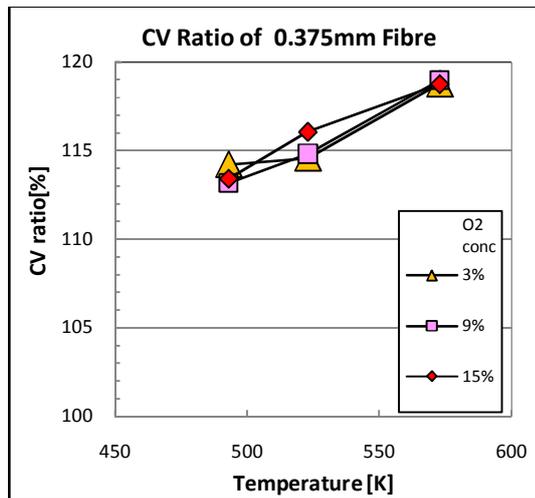


Figure 34: CV Ratio of 0.375mm Fibre

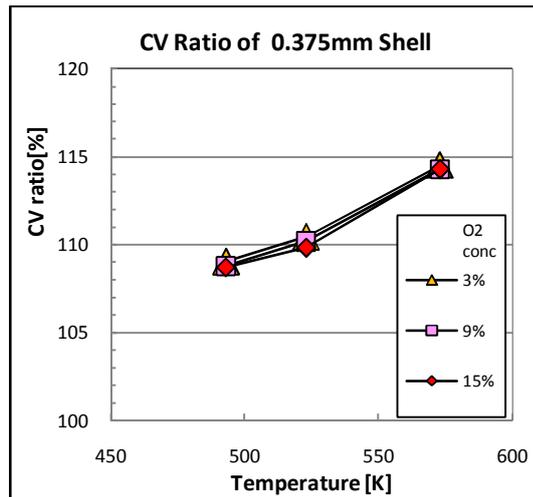


Figure 35: CV Ratio of 0.375mm Shell

4.5 Effect of Particle Size on Energy Yield

The energy yield is the key parameter to understand how much energy has been reserved after torrefaction. Since the effect of particle size on mass yield is not significant, therefore, the energy yield exhibits similar behaviour as the calorific value.

4.6 Effect of Temperature and Oxygen Concentration on Energy Yield

Figure 45 shows the relationship between energy yield and temperature at different oxygen concentrations. For EFB, at 0, 3 and 9 % of oxygen concentrations, the energy yield at 493K is almost the same as that at 573 K. However, torrefaction at 493K is preferable because of its larger calorific value than at 493K.

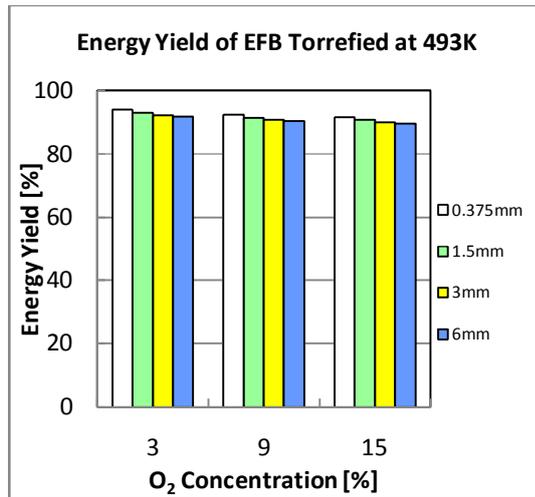


Figure 36: Energy Yield of EFB Torrefied at 493K

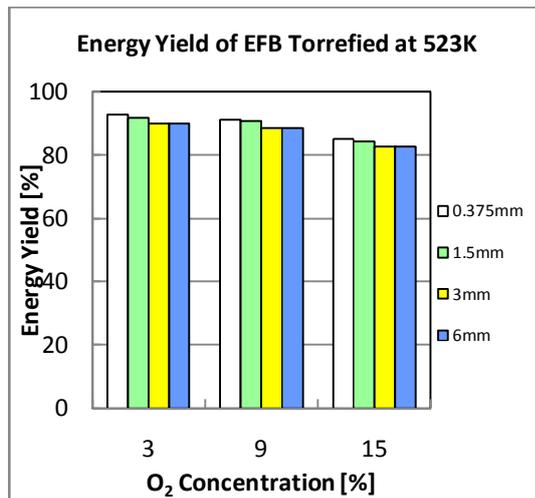


Figure 37: Energy Yield of EFB Torrefied at 523K

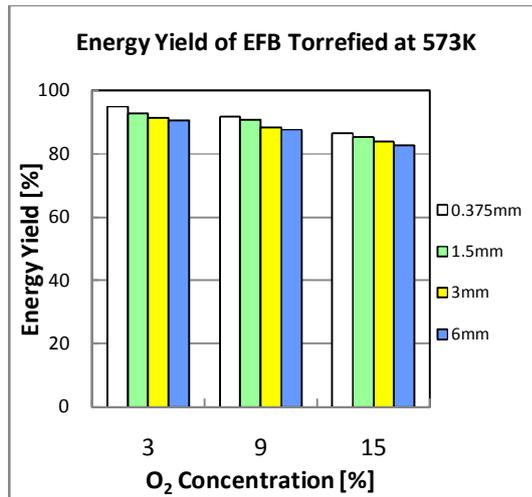


Figure 38: Energy Yield of EFB Torrefied at 573K

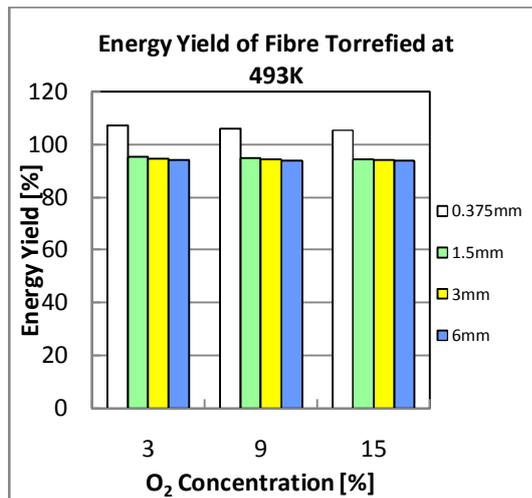


Figure 39: Energy Yield of Fibre Torrefied at 493K

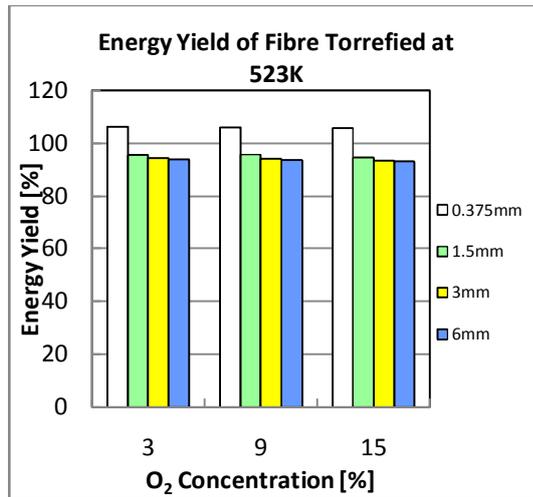


Figure 40: Energy Yield of Fibre Torrefied at 523K

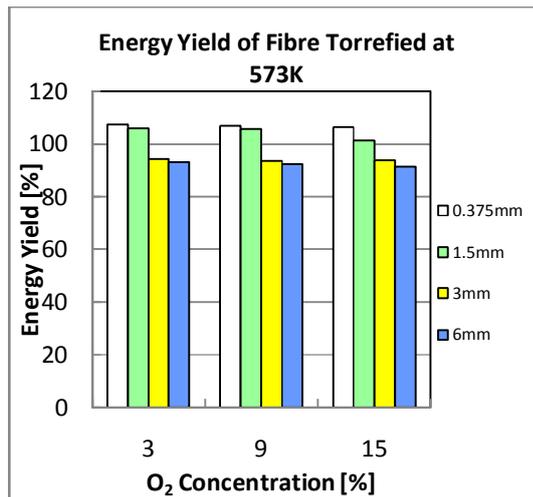


Figure 41: Energy Yield of Fibre Torrefied at 573K

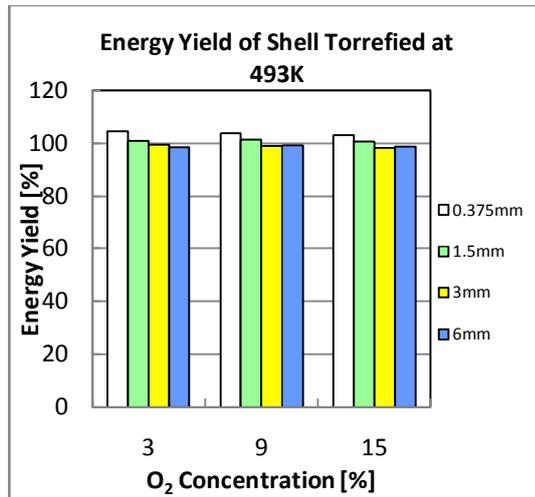


Figure 42: Energy Yield of Shell Torrefied at 493K

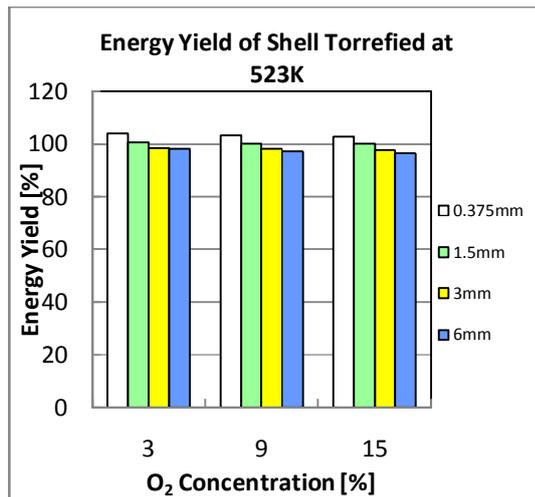


Figure 43: Energy Yield of Shell Torrefied at 523K

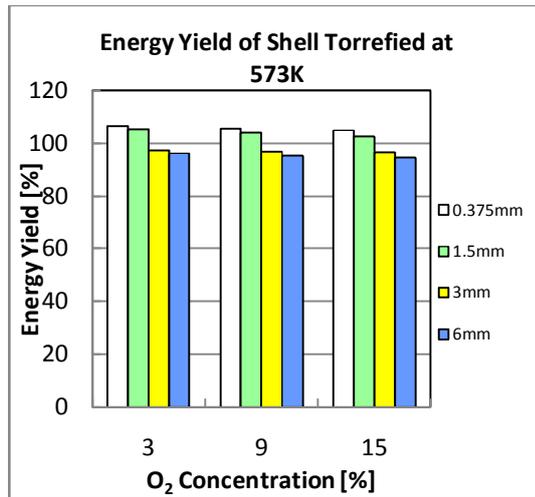


Figure 44: Energy Yield of Shell Torrefied at 573K

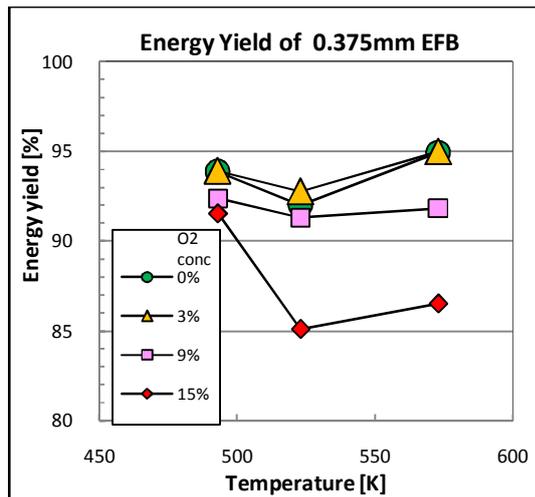


Figure 45: Energy Yield of 0.375mm EFB

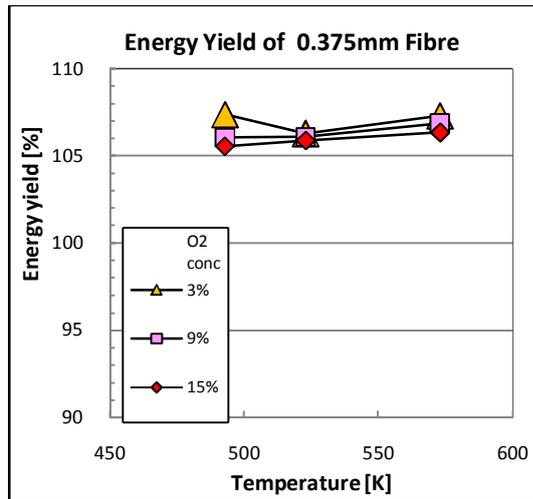


Figure 46: Energy Yield of 0.375mm Fibre

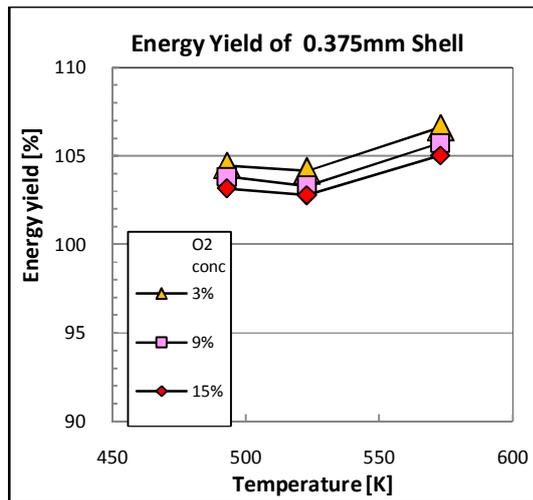


Figure 47: Energy Yield of 0.375mm Shell

4.7 Ultimate Analysis

From ultimate analysis, the value of fixed carbon content can be obtained. This value can be used to verify the calorific value of the biomass. Table 8 shows the result of ultimate analysis of EFB of size 0.375mm and Figure 16 shows effect of temperature and Oxygen concentration to Carbon content. The figure shows no variation of Carbon content regardless of temperature and Oxygen concentration. This behavior does not correlate with the calorific values which showing trend as expected from literature. No report is available to explain this incident.

Author believes that something might have gone wrong during the measurement of the ultimate analysis. One of the possibilities is that the EFB used as sample during the test was not representative. The amount of sample used in the analysis is $\pm 2.000\text{mg}$. Therefore, the chances to pick up the low carbon content EFB are very high. Another observation made by author that can also explain this problem was, after the torrefaction, there was still some part of the EFB that does not torrefied. This might be due to short retention time (30minutes) provided the diameter of the reactor is only 46mm. since torrefaction is gas-solid reaction which is very slow, more time is required to complete the torrefaction.

As for the calorific value, the amount used for the analysis was $\pm 0.500\text{g}$ which may balance between the torrefied and untorrefied EFB. However, it would be very convenient if the research can be carried out for the next time with more proper handling to obtain more accurate results.

Table 10: Results of Ultimate Analysis and Calorific Value of EFB (0.375mm)

Temp [°C]	O ₂ Conc. [%]	Calorific Value [MJ/kg]	Carbon	Hydrogen	Nitrogen
220	0	17.90	46.91	6.69	1.07
250	0	18.20	46.31	6.90	1.43
300	0	20.83	46.47	6.26	1.48
220	3	17.83	46.54	6.75	1.17
250	3	18.23	45.39	6.62	1.35
300	3	20.85	46.55	6.14	1.47
220	9	17.83	46.96	6.35	1.26
250	9	18.21	46.48	6.46	1.40
300	9	20.84	46.30	6.11	1.50
220	15	17.83	46.47	6.23	1.10
250	15	18.23	45.80	6.15	1.53
300	15	20.83	46.65	6.08	1.44

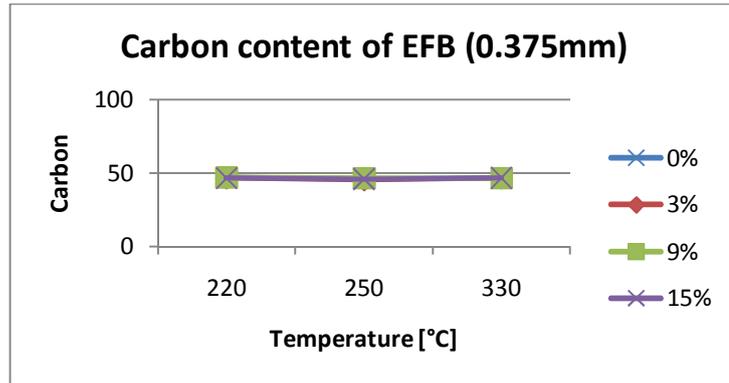


Figure 48: Effect of Temperature and Oxygen Concentration on Carbon content of EFB (0.375mm)

CHAPTER 5

5.0 CONCLUSION AND RECOMMENDATION

EFB was torrefied in the presence of Oxygen to study the effects of torrefaction variables, such as, oxygen concentration (0, 3, 9 and 15 %), temperature (220, 250 and 300 °C) and biomass size (0.375, 1.5, 3 and 6 mm), on the mass and energy yields. The mass yield decreased with an increase in temperature and oxygen concentration, but the effect of biomass size was not significant. The energy yield decreased with an increase in oxygen concentration, but all the values fell within 85 to 95 %. Besides, a similar energy density is obtained from the lowest torrefaction temperature, 220°C, or the highest temperature, 300°C. However, the higher the temperature, the higher the energy density. Therefore, the highest temperature is recommended.

Other than that, it was found that this torrefaction may be divided into two parts; one is ordinary torrefaction, another is oxidation. However, it is worthwhile to carry out torrefaction in the presence of oxygen without any significant problem, while a maximum 7% of biomass is lost by complete oxidation at 15 % of oxygen.

Meanwhile, for the ultimate analysis which shows unexpected results, it would be best if the future research work may consider longer retention time and different configuration of the reactor to be vertical so that the torrefaction gas may pass through the whole biomass. Therefore, better torrefaction might be achieved.

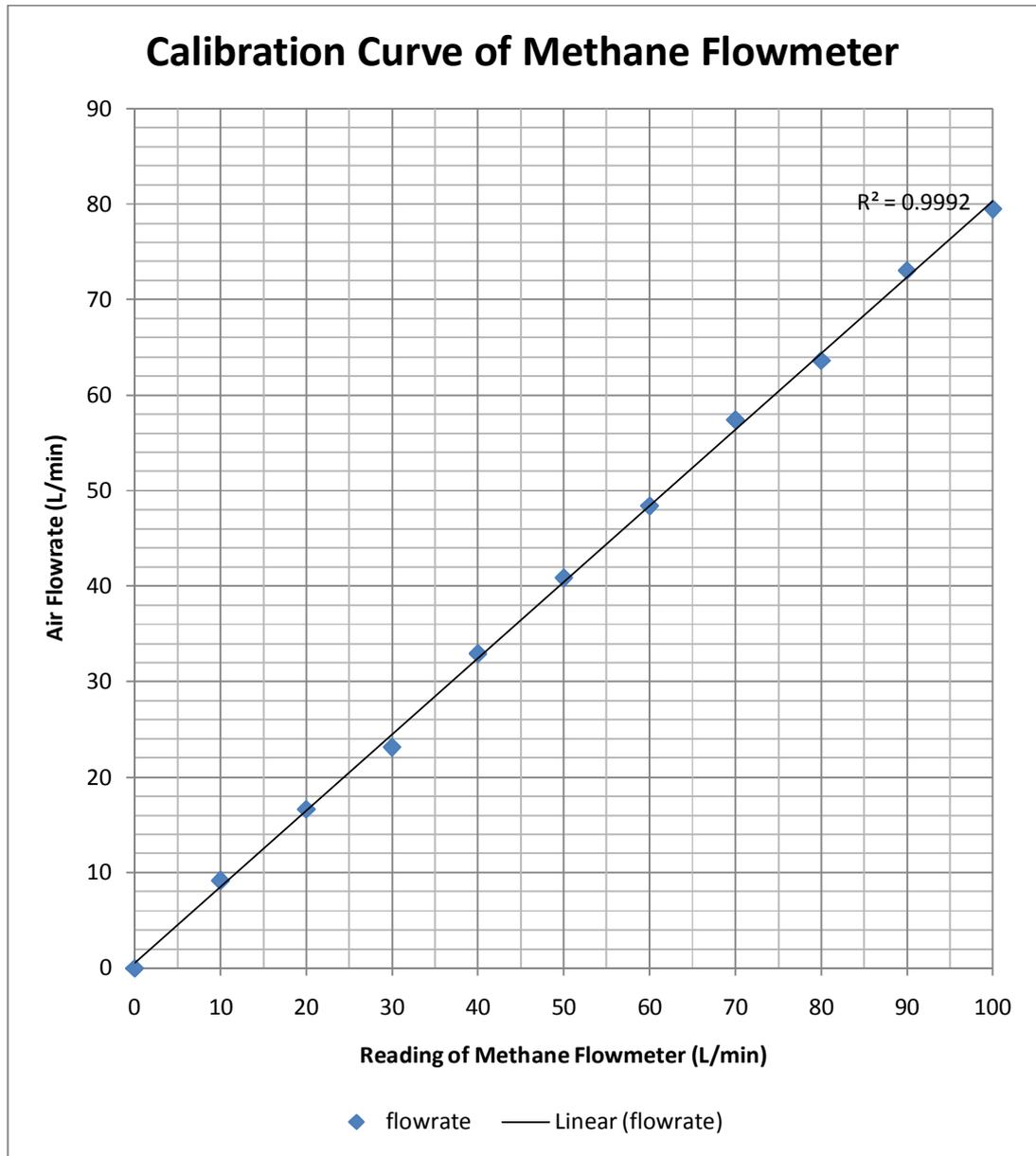
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APPENDICES

Appendix 1: Calibration Curve of Methane Flowmeter



Appendix 2: Raw data of EFB Torrefaction

Temperature (°C)	O ₂ concentration (%)	Size (mm)	Calorific Value (MJ/kg)	Cal Yield	m before	m after	Mass yield	Energy yield
220	3	0.375	17.829	102.36	1.5582	1.4308	91.82	93.9906
220	3	1.5	17.644	101.298	1.5937	1.4639	91.86	93.04726
220	3	3	17.485	100.385	1.4726	1.353	91.88	92.23173
220	3	6	17.433	100.086	1.6073	1.4769	91.89	91.96615
220	9	0.375	17.832	102.377	1.6114	1.455	90.29	92.44031
220	9	1.5	17.64	101.275	1.5259	1.3791	90.38	91.53137
220	9	3	17.494	100.436	1.6497	1.4914	90.40	90.79878
220	9	6	17.431	100.075	1.7112	1.5475	90.43	90.50111
220	15	0.375	17.829	102.36	1.5448	1.3828	89.51	91.62538
220	15	1.5	17.644	101.298	1.5555	1.3946	89.66	90.81935
220	15	3	17.485	100.385	1.4935	1.3395	89.69	90.03365
220	15	6	17.433	100.086	1.4772	1.3255	89.73	89.80785
250	3	0.375	18.229	104.656	1.6692	1.4807	88.71	92.83746
250	3	1.5	18.016	103.433	1.6464	1.462	88.80	91.84851
250	3	3	17.673	101.464	1.6914	1.5023	88.82	90.12024
250	3	6	17.67	101.447	1.5432	1.3716	88.88	90.16615
250	9	0.375	18.211	104.553	1.7654	1.543	87.40	91.38151
250	9	1.5	18.101	103.921	1.7785	1.5549	87.43	90.85585
250	9	3	17.671	101.453	1.6543	1.4465	87.44	88.70886
250	9	6	17.663	101.407	1.4582	1.275	87.44	88.66644
250	15	0.375	18.232	104.673	1.5538	1.2645	81.38	85.18434
250	15	1.5	18.016	103.433	1.436	1.1722	81.63	84.43206
250	15	3	17.669	101.441	1.5543	1.2698	81.70	82.87321
250	15	6	17.669	101.441	1.5991	1.3067	81.71	82.89225
300	3	0.375	20.85	119.704	1.7121	1.3597	79.42	95.06524
300	3	1.5	20.351	116.839	1.6632	1.3232	79.56	92.95409
300	3	3	20.007	114.864	1.6834	1.3413	79.68	91.52132
300	3	6	19.809	113.727	1.5472	1.2351	79.83	90.78622
300	9	0.375	20.836	119.623	1.6129	1.2389	76.81	91.88505
300	9	1.5	20.571	118.102	1.6836	1.2949	76.91	90.83525
300	9	3	20.014	114.904	1.7111	1.3174	76.99	88.4663
300	9	6	19.799	113.67	1.5492	1.1946	77.11	87.65163
300	15	0.375	20.833	119.606	1.4917	1.0801	72.41	86.60361
300	15	1.5	20.48	117.58	1.6388	1.189	72.55	85.30757
300	15	3	20.1	115.398	1.5837	1.1531	72.81	84.02177
300	15	6	19.783	113.578	1.6272	1.1874	72.97	82.88004

Appendix 3: Raw data of Fibre Torrefaction

Temperature (°C)	O ₂ concentration (%)	Size (mm)	Calorific Value (MJ/kg)	Cal yield	m before	m after	Mass yield	Energy yield
220	3	0.375	21.25	114.23	2.2127	2.0799	94.00	107.37
220	3	1.5	18.82	101.18	2.2482	2.1174	94.18	95.30
220	3	3	18.67	100.39	2.1271	2.0045	94.24	94.60
220	3	6	18.58	99.87	2.2618	2.1328	94.30	94.18
220	9	0.375	21.05	113.16	2.2659	2.1237	93.72	106.06
220	9	1.5	18.83	101.24	2.1804	2.0443	93.76	94.92
220	9	3	18.69	100.46	2.3042	2.1616	93.81	94.24
220	9	6	18.62	100.12	2.3657	2.2207	93.87	93.98
220	15	0.375	21.10	113.43	2.1993	2.0465	93.05	105.55
220	15	1.5	18.84	101.26	2.21	2.0613	93.27	94.45
220	15	3	18.72	100.66	2.148	2.0064	93.41	94.03
220	15	6	18.63	100.15	2.1317	1.9964	93.65	93.80
250	3	0.375	21.31	114.59	2.3237	2.1553	92.75	106.28
250	3	1.5	19.13	102.82	2.3009	2.1352	92.80	95.42
250	3	3	18.88	101.52	2.3459	2.1788	92.88	94.29
250	3	6	18.78	100.96	2.1977	2.0419	92.91	93.80
250	9	0.375	21.36	114.83	2.4199	2.2356	92.38	106.09
250	9	1.5	19.25	103.49	2.2561	2.0856	92.44	95.67
250	9	3	18.91	101.64	2.3088	2.1383	92.62	94.13
250	9	6	18.78	100.96	2.1127	1.9586	92.71	93.59
250	15	0.375	21.59	116.07	2.2083	2.0141	91.21	105.87
250	15	1.5	19.25	103.50	2.0905	1.9104	91.38	94.58
250	15	3	18.91	101.66	2.2088	2.0306	91.93	93.45
250	15	6	18.81	101.14	2.2536	2.0734	92.00	93.05
300	3	0.375	22.09	118.77	2.3666	2.1379	90.34	107.30
300	3	1.5	21.74	116.88	2.3177	2.0985	90.54	105.82
300	3	3	19.34	103.98	2.3379	2.1213	90.74	94.35
300	3	6	19.02	102.27	2.2017	2.0035	91.00	93.07
300	9	0.375	22.14	119.00	2.2674	2.0362	89.80	106.87
300	9	1.5	21.87	117.58	2.3381	2.1031	89.95	105.76
300	9	3	19.36	104.09	2.3656	2.1288	89.99	93.67
300	9	6	19.07	102.51	2.2037	1.9872	90.18	92.44
300	15	0.375	22.09	118.76	2.1462	1.9216	89.53	106.33
300	15	1.5	21.04	113.13	2.2933	2.0548	89.60	101.36
300	15	3	19.47	104.64	2.2382	2.0069	89.67	93.83
300	15	6	18.97	102.00	2.2817	2.0474	89.73	91.53

Appendix 4: Raw data of Shell Torrefaction

Temperature (°C)	O ₂ concentration (%)	Size (mm)	Calorific Value (MJ/kg)	Cal yield	m before	m after	Mass yield	Energy yield
220	3	0.375	21.97	110.42	3.2821	3.1443	95.80	105.78
220	3	1.5	20.95	105.33	3.3176	3.1807	95.87	100.99
220	3	3	20.63	103.67	3.1965	3.0657	95.91	99.43
220	3	6	20.38	102.43	3.3312	3.2064	96.25	98.59
220	9	0.375	21.92	110.18	3.3353	3.1832	95.44	105.15
220	9	1.5	21.09	105.99	3.2498	3.1066	95.59	101.32
220	9	3	20.63	103.72	3.3736	3.2258	95.62	99.17
220	9	6	20.65	103.80	3.4351	3.2863	95.67	99.30
220	15	0.375	21.85	109.82	3.2687	3.1015	94.88	104.20
220	15	1.5	21.09	106.00	3.2794	3.1142	94.96	100.66
220	15	3	20.59	103.49	3.2174	3.0603	95.12	98.44
220	15	6	20.65	103.78	3.2011	3.0497	95.27	98.87
250	3	0.375	21.69	109.04	3.3931	3.1997	94.30	102.82
250	3	1.5	21.18	106.47	3.3703	3.1849	94.50	100.62
250	3	3	20.70	104.05	3.4153	3.2302	94.58	98.41
250	3	6	20.59	103.49	3.2671	3.094	94.70	98.01
250	9	0.375	21.64	108.77	3.4893	3.2717	93.76	101.99
250	9	1.5	21.20	106.58	3.3205	3.1224	94.03	100.22
250	9	3	20.72	104.17	3.3782	3.1785	94.09	98.01
250	9	6	20.53	103.21	3.1821	2.9958	94.15	97.17
250	15	0.375	21.63	108.74	3.2777	3.0669	93.57	101.75
250	15	1.5	21.31	107.13	3.1599	2.9581	93.61	100.28
250	15	3	20.73	104.18	3.2782	3.0712	93.69	97.60
250	15	6	20.49	103.00	3.323	3.1092	93.57	96.38
300	3	0.375	22.78	114.53	3.436	3.1977	93.06	106.59
300	3	1.5	22.51	113.13	3.3871	3.1558	93.17	105.40
300	3	3	20.79	104.52	3.4073	3.1785	93.29	97.50
300	3	6	20.48	102.93	3.2711	3.0551	93.40	96.14
300	9	0.375	22.74	114.30	3.3368	3.086	92.48	105.71
300	9	1.5	22.37	112.43	3.4075	3.1533	92.54	104.04
300	9	3	20.85	104.79	3.435	3.1812	92.61	97.05
300	9	6	20.43	102.70	3.2731	3.0366	92.77	95.28
300	15	0.375	22.74	114.28	3.2156	2.9552	91.90	105.03
300	15	1.5	22.21	111.64	3.3627	3.0915	91.94	102.64
300	15	3	20.89	105.03	3.3076	3.0463	92.10	96.73
300	15	6	20.43	102.67	3.3511	3.0902	92.21	94.68

Appendix 5: CHNS Raw Data for EFB of 0.375mm

Temperature (°C)	O ₂ Concentration (%)	C (%)	H (%)	N (%)
220	0	46.96	6.49	1
220	0	46.86	6.89	1.14
		46.91	6.69	1.07
220	3	46.38	6.77	1.19
220	3	46.69	6.72	1.15
		46.535	6.745	1.17
220	9	46.91	6.31	1.24
220	9	47.01	6.39	1.28
		46.96	6.35	1.26
220	15	46.29	6.2	1.03
220	15	46.64	6.26	1.17
		46.465	6.23	1.1
250	0	46.38	7.02	1.42
250	0	46.23	6.77	1.44
		46.305	6.895	1.43
250	3	45.45	6.65	1.41
250	3	45.33	6.58	1.28
		45.39	6.615	1.345
250	9	46.17	6.43	1.34
250	9	46.79	6.48	1.45
		46.48	6.455	1.395
250	15	45.63	6.2	1.58
250	15	45.96	6.09	1.47
		45.795	6.145	1.525
300	0	46.23	6.23	1.44
300	0	46.71	6.28	1.51
		46.47	6.255	1.475
300	3	46.64	6.17	1.35
300	3	46.45	6.1	1.58
		46.545	6.135	1.465
300	9	46.06	6.12	1.58
300	9	46.53	6.09	1.42
		46.295	6.105	1.5
300	15	46.79	6.12	1.39
300	15	46.51	6.03	1.49
		46.65	6.075	1.44