CHARACTERIZATION OF BIOCHAR DERIVED FROM TAPIOCA WASTES

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CHARACTERIZATION OF BIOCHAR DERIVED FROM TAPIOCA WASTES

by

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LIST OF ABBREVIATIONS

AC	Ash content
AIM	Agensi Inovasi Malaysia
AMF	Arbuscular mycorrhizal fungi
APN	Asia-Pacific Network for Global Change Research
ASTM	American Society for Testing and Materials
BET	Brunauer, Emmett and Teller
BPA	Biomass Power Association
BSE	Backscattered electrons
$C_2H_4O_2$	Acetic acid
$C_6H_{12}O_6$	Glucose
CEC	Cation exchange capacity
CH_4	Methane
CO	Carbon monoxide
CO_2	Carbon dioxide
DOSM	Department of Statistics Malaysia
DTG	Derivative thermogravimetry
EDX	Energy-dispersive X-ray
EFB	Empty fruit bunch
FAO	Food and Agriculture Organization of the United Nations
FC	Fixed carbon
GHG	Greenhouse gases
GNI	Gross National Income
H_2	Hydrogen
H_2O	Water
HCl	Hydrochloric acid
IBI	International Biochar Initiative
IEA	International Energy Agency
MF	Mesocarp fibre
N_2	Nitrogen
$NaClO_2$	Sodium chlorite

NaOH	Sodium hydroxide
NASA	National Aeronautics and Space Administration
NBS	National Biomass Strategy
NMR	Nuclear magnetic resonance
NO_x	Nitrogen oxides
O_2	Oxygen
OECD	Organisation for Economic Co-operation and Development
OPF	Oil palm fronds
OPT	Oil palm trunk
PIK	Potsdam Institute for Climate Impact Research
PKS	Palm kernel shell
POME	Palm oil mill effluent
SEM	Scanning electron microscopy
SIO	Scripps Institution of Oceanography
SO_2	Sulphur dioxide
SOM	Soil organic matter
SO_x	Sulphur oxides
TAS	Thai Agricultural Standard
TG	Thermogravimetry
TRB	Tapioca rhizome biochar
TSB	Tapioca stem biochar
VM	Volatile matter
WRI	World Resources Institute

LIST OF SYMBOLS

%	percent
°C	degree Celsius
cal/cm	calories per centimeter (1 cal = 4.19 J)
cm	centimeter
dwMmtons	dry weight million metric tons
g	gram
h	hour
ha	hectare
J	joules
K	Kelvin
L	liters
m	meter
mf wt. %	weight percent on moisture free basis
min	minute
MMT	million metric tons (1 metric ton = 1 tonne = 1000 kg)
Mt	million tons (1 ton = 907.185 kg)
Mtoe	million tons of oil equivalents (1 Mtoe = 11.63 terawatt hour)
Pa	Pascal
ppm	parts per million (for concentration of CO_2 gas, 1 ppm = 1.94 mg/m^3)
s	second
t/ha	metric tons per hectare
wt. %	weight percent

PENCIRIAN BIOARANG TERHASIL DARI PIROLISIS PERLAHAN SISA-SISA UBI KAYU

ABSTRAK

Objektif utama kajian ini adalah untuk menyiasat kesan pelbagai parameter pirolisis seperti suhu terminal, kadar pemanasan, masa tetapan dan kandungan abu bahan suapan terhadap hasil bioarang dan ciri-cirinya. Bioarang telah dihasilkan dengan menggunakan bahan suapan batang ubi kayu dan rhizome ubi kayu melalui proses pirolisis perlahan berskala makmal. Ciri-ciri bioarang ditentukan dengan menggunakan analisis proksimat, analisis unsur, analisis Mikroskop Imbasan Elektron (SEM) dan analisis luas permukaan Brunauer, Emmett dan Teller (BET). Sebelum eksperimen, ciri-ciri bahan suapan telah ditentukan dan didapati bahawa kedua-dua batang ubi kayu dan rhizome ubi kayu mengandungi peratusan bahan meruap dan nilai kalori yang agak tinggi. Ini menunjukkan bahawa kedua-dua bahan suapan adalah sesuai untuk melalui proses termokimia seperti pirolisis perlahan. Kedua-dua bahan suapan juga didapati menjadi bahan suapan mesra alam kerana ia mengandungi kandungan nitrogen dan sulfur yang rendah. Kandungan lignin dan selulosa yang tinggi dalam kedua-dua batang ubi kayu dan rhizome ubi kayu menunjukkan bahawa bahan suapan ini adalah sesuai bagi penghasilan bioarang. Peningkatan suhu terminal pirolisis mengurangkan hasil bioarang batang ubi kayu (TSB) dan bioarang rhizome ubi kayu (TRB). Apabila suhu terminal meningkat dari 400 °C hingga 650 °C, hasil TSB berkurangan dari 35.86 mf wt. % kepada 25.79 mf wt. % manakala hasil TRB menurun dari 36.98 mf wt. % kepada 28.82 mf wt. %. Bioarang yang dihasilkan pada suhu terminal yang lebih tinggi meningkatkan kandungan karbon tetapnya. Apabila suhu terminal pirolisis meningkat dari 400 °C hingga 650 °C, kandungan karbon tetap TSB yang dihasilkan meningkat dari 78.39 mf wt. % kepada 87.88 mf wt. % manakala kandungan karbon tetap TRB meningkat dari 79.20 mf wt. % kepada 84.46 mf wt. %. Peningkatan suhu terminal dari 400 °C hingga 600 °C meningkatkan luas permukaan TSB dari 0.83 m^2/g

hingga 36.12 m^2/g dan luas permukaan TRB juga meningkat dari 0.24 m^2/g kepada 12.29 m^2/g. Parameter kadar pemanasan tidak memberi kesan yang ketara ke atas hasil bioarang dan komposisinya. Walau bagaimanapun, kadar pemanasan yang lebih tinggi menghasilkan lebih banyak liang-liang pada permukaan TSB dan TRB sekaligus meningkatkan luas permukaannya. TSB yang dihasilkan pada kadar pemanasan 25 °C/min mempunyai luas permukaan 10.58 m^2/g iaitu 12 kali lebih besar daripada luas permukaan TSB yang dihasilkan pada kadar pemanasan 5 °C/min. Luas permukaan TRB yang dihasilkan pada kadar pemanasan 25 °C/min didapati adalah 10.98 m^2/g iaitu 45 kali lebih besar daripada luas permukaan TRB yang dihasilkan pada kadar pemanasan 5 °C/min. Hasil TSB dan TRB menurun apabila masa tetapan pirolisis meningkat. Apabila masa tetapan dilanjutkan dari 1.0 jam kepada 3.5 jam, hasil TSB berkurang dari 35.86 mf wt. % kepada 33.27 mf wt. % manakala hasil TRB juga menurun dari 36.98 mf wt. % kepada 32.23 mf wt. %. Peningkatan masa tetapan dari 1.0 jam kepada 3.5 jam meningkatkan kandungan karbon tetap TSB dari 78.39 mf wt. % kepada 81.88 mf wt. % dan meningkatkan kandungan karbon tetap TRB dari 79.20 mf wt. % kepada 83.29 mf wt. %. Kesan masa tetapan terhadap morfologi permukaan bioarang adalah lebih ketara pada TRB berbanding TSB. Peningkatan masa tetapan dari 1.0 jam kepada 3.0 jam meningkatkan kawasan permukaan TSB dan TRB masing-masing sebanyak 5 kali dan 29 kali ganda. Pra-rawatan basuhan air mengurangkan kandungan abu bahan suapan rhizome ubi kayu dari 7.28 mf wt. % kepada 2.24 mf wt. %. Kesan kandungan abu bahan suapan rhizome ubi kayu adalah tidak jelas pada ciri-ciri bioarang yang dihasilkan, tetapi bahan suapan dengan kandungan abu yang rendah telah didapati menurunkan hasil bioarang. Pengurangan kandungan abu bahan suapan rhizome ubi kayu dari 7.28 mf wt. % kepada 2.24 mf wt. % menurunkan hasil bioarang dari 36.98 mf wt. % kepada 31.83 mf wt. %.

CHARACTERIZATION OF BIOCHAR DERIVED FROM TAPIOCA WASTES

ABSTRACT

The main objective of this study is to investigate the impacts of various pyrolysis parameters such as terminal temperature, heating rate, holding time and ash content of feedstock on the biochar yield and its characteristics. The biochars were produced using tapioca stem and tapioca rhizome feedstocks via laboratory scale slow pyrolysis process. The biochars were characterized using the proximate analysis, ultimate analysis, Scanning Electron Microscope (SEM) analysis and Brunauer, Emmett and Teller (BET) surface area analysis. Prior to the experiment, the feedstocks have been characterized and it was found that both tapioca stem and tapioca rhizome contained fairly high percentage of volatile matter and calorific values. This indicated that both feedstocks are suitable to undergo the thermochemical process such as slow pyrolysis. Both feedstocks were also found to be the environmental friendly feedstocks as it contains low nitrogen and sulphur content. High lignin and cellulose contents in both tapioca stem and tapioca rhizome indicated that the feedstocks were suitable for the biochar production. The increment of pyrolysis terminal temperature had significantly reduced the yield of tapioca stem biochar (TSB) and tapioca rhizome biochar (TRB). As the terminal temperature increased from 400 °C to 650 °C, the yield of TSB had decrease from 35.86 mf wt. % to 25.79 mf wt. % while the yield of TRB decreased from 36.98 mf wt. % to 28.82 mf wt. % respectively. Biochar produced at higher terminal temperature had higher fixed carbon content. As the pyrolysis terminal temperature rose from 400 °C to 650 °C, the TSB produced increased in fixed carbon content from 78.39 mf wt. % to 87.88 mf wt. % while the fixed carbon content of TRB increased from 79.20 mf wt. % to 84.46 mf wt. %. The increment of terminal temperature from 400 °C to 600 °C had increase the surface area of TSB from 0.83 m^2/g to 36.12 m^2/g and the surface area of TRB also increased from 0.24 m^2/g to 12.29 m^2/g. The heating rate parameter did not give pronounced effect on the biochar yield and its composition. However, higher heating rate developed more pores on the surface of TSB and TRB thus increased its surface areas. The TSB produced at a heating rate of 25 °C/min had the surface area of 10.58 m^2/g, which is 12 times larger than the surface area of the TSB produced at a heating rate of 5 °C/min. The surface area of TRB produced at a heating rate of 25 °C/min was found to be 10.98 m^2/g, which is 45 times larger than the surface area of TRB produced at a heating rate of 5 °C/min. The yield of TSB and TRB decreased as the holding time of pyrolysis was increased. As the holding time was prolonged from 1.0 hours to 3.5 hours, the yield of TSB reduced from 35.86 mf wt. % to 33.27 mf wt. % while the yield of TRB also reduced from 36.98 mf wt. % to 32.23 mf wt. %. The increment of holding time from 1.0 hours to 3.5 hours had increase the fixed carbon content of TSB from 78.39 mf wt. % to 81.88 mf wt. % and increased the fixed carbon content of TRB from 79.20 mf wt. % to 83.29 mf wt. %. The effect of holding time on the surface morphology of biochar was more apparent on the TRB compared to the TSB. The increment of holding time from 1.0 hours to 3.0 hours increased the surface area of TSB and TRB by 5 times and 29 times respectively. The water washing pre-treatments reduced the ash content of the tapioca rhizome feedstock from 7.28 mf wt. % to 2.24 mf wt. %. The effect of ash content of the tapioca rhizome feedstock was not apparent on the characteristics of the biochar produced, but feedstock with lower ash content was found to decrease the biochar yield. The reduction of ash content of the tapioca rhizome feedstock from 7.28 mf wt. % to 2.24 mf wt. % decreased the biochar yield from 36.98 mf wt. % to 31.83 mf wt. %.

CHAPTER 1

INTRODUCTION

1.1 Sustainable Biochar to Mitigate Global Climate Change

The energy demands of modern societies have been steadily increased each year. The main source of energy in worldwide is fossil fuel and the utilization of fossil fuel became the primary source of carbon dioxide (CO_2) emission (Bernstein et al., 2007). CO_2 is the primary source of greenhouse gases (GHG), while GHG is the major cause of global warming that led to the world's climate change (OECD/IEA, 2013). Common renewable energy strategies can at best off set fuel emissions of CO_2, but not able to reverse the climate change. One promising approach of lowering CO_2 in atmosphere is the biochar production in combination with its utilization as the soil amendment (Lehmann, 2007, Woolf et al., 2010, Cushion et al., 2010).

The International Biochar Initiative (IBI) defines biochar as "the solid material obtained from the carbonization of biomass that may be added to soils with the intention to improve soil functions and to reduce emissions from biomass that would otherwise naturally degrade to GHG" (IBI, 2013b). Biochar was produced by means of thermochemical conversion process. So far, pyrolysis is the most important biomass conversion process, in the context of biochar production (Biofuelwatch, 2011, Lehmann et al., 2006). By using various types of reactor configuration, pyrolysis involves the heating process of biomass at moderate temperature, under complete or partial exclusion of oxygen (Lehmann, 2007, Woolf, 2008).

Biochar should be made from biomass waste materials to avoid the competition with any other land use. The abundant of wastes can also be reduced if it were used as the feedstock for the biochar production. The appropriate biomass for biochar production including the crop residues, forestry wastes, animal manures and food wastes (IBI, 2013a).

As biomass or organic matter decay, the GHG such as methane (CH_4) and CO_2 were released into the atmosphere, where CH_4 is known to be 21 times more potent GHG than CO_2. However, by charring the biomass, many of its carbon content becomes 'fixed' into a more stable form (Hunt et al., 2010). Consequently, carbon content in the produced biochar had greater longevity compared to its raw biomass (Lehmann et al., 2006). Figure 1.1 shows the comparison of carbon remaining in the biochar and un-charred organic matter.

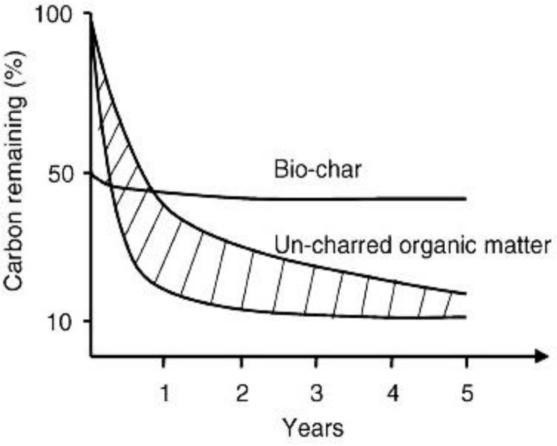


Figure 1.1: Carbon remaining in biomass and biochar after decomposed in the soil (Lehmann et al., 2006)

Evidence suggested that component of carbon in biochar was highly recalcitrant or very stable in soils. The residence time for wood biochar was reported to be in the range from 100 years to 1,000 years. Biochar components decomposed very slowly and allowed the carbon input into soil to be increased greatly compared to the carbon output, which led to the biochar's possible carbon negativity (Verheijen et al., 2009).

Figure 1.2 represents an overview of sustainable biochar concept. The figure shows that 50 % of the pyrolyzed biomass was converted into biochar and applied to soil; means that half of the original carbon retained in the biochar was returned to soil. Meanwhile, the remaining carbon in biomass was converted into the bio-fuels and heat. (Cushion et al., 2010, Lehmann, 2007, Gaunt and Driver, 2010).

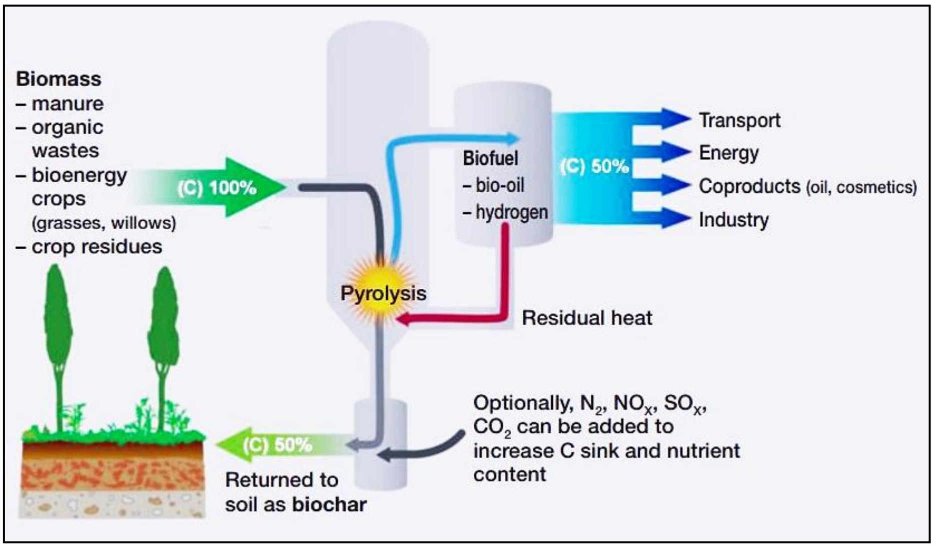


Figure 1.2: The overview of sustainable biochar concept
(Lehmann, 2007)

It is important to know the characteristics of biochar in order to determine whether it influence the soil characteristics and remain permanent in the soil. Understanding the basic properties of biochar was also beneficial for identifying their appropriate applications and for upgrading them. From the literature review, it can be concluded that not all biochar was created equal. There are many factors that influenced the key physical and chemical characteristics of biochar. For example, the type of biomass feedstock, ash content of feedstock and the conditions during the production process including the pyrolysis terminal temperature, heating rate and holding time, as discussed further in Section 2.3.3.

1.2 Tapioca Wastes for Biochar Production

Tapioca (*Manihot esculenta* Crantz) or also known as cassava was cultivated worldwide mainly for its starchy tuberous roots. Tapioca was the third largest source of carbohydrates for human consumption around the world (Kuiper et al., 2007). According to Food and Agriculture Organization of the United Nations (FAO), there was high demand on tapioca and its global production had doubled from the past 30 years to over 262 million tons in year 2012 (FAO, 2014).

The disposal of leftover tapioca in the field had raised concerns. Tapioca wastes such as the stem and the rhizome which was not edible for human was kept aside and accumulated every year in large quantities. Tapioca wastes can be directly used for energy production via direct combustion process. However, Kauffman et al. (1995) stated that about 50 % of carbon in the biomass was lost directly upon burning. The better option is by converting the tapioca wastes into biochar via greener and sustainable pyrolysis process. This approach can be made to clean the tapioca field and at the same time preserved the carbon content.

1.3 Problem Statement

Malaysia is a tropical country and blessed with various types of plants and agriculture. The agricultural sector such as tapioca produced wastes which are mostly dumped in the plantation or pile areas instead of being used as by-products. The land fill of this organic wastes resulted in the release of significant quantities of CH_4 (Kwapinski et al., 2010). As an alternative, tapioca wastes could be used as the feedstock to produce biochar and therefore help minimize the waste management problem.

The study of biochar characterization is necessary because of their potential environmental applications including as the soil amendment and for the atmospheric carbon sequestration. Currently, a number of studies have been carried out in Malaysia to characterize the biochar produced from different biomass especially oil palm wastes under different conditions. However, there is insufficient data on biochar characteristics produced from other types of biomass including the tapioca wastes. Thus, the characterization of biochar produced from tapioca wastes would act as a fundamental study for biochar applications. This research will provide deeper understanding into the properties of biochar produced from tapioca wastes using various production parameters of terminal temperature, heating rate, holding time and feedstock ash content. This study also can be one of the initiatives to ensure the sustainable development since biochar is considered as a potential tool to mitigate the climate change.

1.4 Research Objectives

The objectives of the present study are:

i) To identify the characteristics of tapioca stem and tapioca rhizome as the feedstock for biochar production via slow pyrolysis.

ii) To analyze the effects of pyrolysis parameters including the terminal temperature, heating rate and holding time on the yield and characteristics of biochar produced from tapioca stem and tapioca rhizome feedstock.

iii) To investigate the impact of ash content of tapioca rhizome feedstock on the biochar yield and its characteristics.

1.5 Scope of Research

This research studies the characteristics of two types of tapioca wastes which are tapioca stem and tapioca rhizome as the feedstock for biochar production. Both tapioca wastes were converted into biochars by slow pyrolysis process using different parameters. The studied parameters are the terminal temperature, heating rate, holding time and ash content of the feedstock. We also study the percentage yield of biochar as the function of the different parameters. The impacts of the various parameters on the characteristics of biochars were also being analyzed.

1.6 Thesis Outline

The thesis was arranged into the following chapters:

Chapter 2 described the overview of biomass in Malaysia and detailed information of lignocellulosic biomass including the tapioca wastes. Information about pyrolysis conversion processes including slow pyrolysis, fast pyrolysis, flash pyrolysis and hydropyrolysis were also explained in this chapter. This chapter also reviews the past pyrolysis experiments for biochar production from various lignocellulosic biomass. The focus of the reviews was more on the effects of pyrolysis parameters and ash content of the feedstock on yield and characteristics of the biochar produced. The properties of biochar and results of using biochar as the soil amendment and as a tool to mitigate climate change were also reviewed in more detail within this chapter.

Chapter 3 described the preparation of feedstock and the methods used to characterize the feedstock prior to the slow pyrolysis. This chapter elaborated the experimental set-up and the procedures of slow pyrolysis experiment for this study. The experimental parameters of slow pyrolysis included the terminal temperature, heating rate and holding time were varied to study the impacts on the biochar production. The varied parameters were explained further in this chapter. This chapter also described the method of water washing pre-treatment used on the tapioca rhizome to reduce its ash content. The ash content of tapioca rhizomes was reduce in order to study the effects of ash content of the feedstock on the biochar production. The analyses used to determine the chemical and physical properties of the biochars produced were also been described further in this chapter.

Chapter 4 had basically divided into two main parts that separate the results of characteristics of the feedstock and the results of biochar production via slow pyrolysis in various parameters. The biochar production results include the percentage yield and characteristics of the biochars.

Finally, Chapter 5 provided a conclusion remark based on the work presented in the previous chapters. It also included some recommendations for the future study.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview of Biomass

2.1.1 Introduction

Biomass means any organic matter that is renewable. It stored energy that produced during photosynthesis (Ashton et al., 2002). Photosynthesis is a process that involved living plant, carbon dioxide (CO_2), water (H_2O), heat energy from sunlight and chlorophyll from the plant as the catalyst and, in order to produce glucose ($C_6H_{12}O_6$), oxygen (O_2) and chemical energy. The chemical energy produced from photosynthesis is stored in plants and then passed on to the animals or human that consumes the plant. Equation 2.1 represents the chemical reaction of photosynthesis process that produce 480 kJ/mol of chemical energy (Hodge, 2010).

$$\text{Living plant} + 6CO_2 + 6H_2O + \text{Sunlight} \xrightarrow{\text{Chlorophyll}} C_6H_{12}O_6 + 6O_2 - 480\ \text{kJ/mol} \quad (2.1)$$

Biomass comes from a variety of sources and can be divided into two broad groups which are primary biomass and derived biomass or waste. Table 2.1 shows the sub-classification of primary biomass and waste. A major part of biomass is lignocellulosic biomass includes wood, plants and leaves that were described in more detail in Section 2.1.3.

Table 2.1: Two major groups of biomass and their sub-classifications (Basu, 2010)

Group of biomass	Sub-classification	Examples
Primary biomass	Terrestrial biomass	Forest biomass Grasses Energy crops
	Aquatic biomass	Algae Water plant
Wastes	Municipal wastes	Municipal solid wastes Biosolids, sewage

	Agricultural solid wastes	Livestock and manures Agricultural crop residues
	Forestry residues	Bark, leaves, floor residues
	Industrial wastes	Demolition wood, sawdust Waste oil or fat

2.1.2 Biomass in Malaysia

International Energy Agency (IEA) reported that in year 2010, the Total Primary Energy Supply in Malaysia is 72.6 million ton of oil equivalents (Mtoe), of which 5.5 % or 4.0 Mtoe was produced from renewable energy sources. 86 % of renewable energy sources in Malaysia are from biomass and biofuels while the remaining 14 % is from hydro energy (IEA, 2012). The most abundant biomass or renewable wastes in Malaysia are agricultural residues from the agricultural sector.

Agricultural sector is one of the primary pillars of the national economy in Malaysia. Agensi Inovasi Malaysia (AIM) had reported that, Malaysia currently generates about 12 % of Gross National Income (GNI) from the agriculture sector (AIM, 2013). The agriculture land in Malaysia is mostly plantation with more than 77 % is industrial crops such as oil palm, rubber, cocoa, pineapple and pepper, while the remaining land is under annual food crops such as paddy, vegetables, fruits and others (Agamuthu and Fauziah, 2010). The agricultural activities create a tremendous amount of organic waste materials. The climate in Malaysia which is high in sunlight intensity and rainfall make the production of agricultural residues from plantations is throughout the year (Zwart, 2013). The production of agricultural residues in Malaysia was estimated to be more than 70 million tons (Mt) per year (Agamuthu and Fauziah, 2010).

Within agriculture sectors, by far the palm oil sector constitutes the largest GNI, contributing about 8 % or over RM 80 billions (AIM, 2013). Correspondingly, the palm oil sector generates the largest portion of biomass with gross value of 85 % of agricultural residues discharged per year (Agamuthu and Fauziah, 2010). The estimated amount of biomass from oil palm sector is about 80 dry weight million metric tons (dwMmtons) per year. In year 2012, Malaysia's palm oil sector produced over 83 dwMmtons of solid biomass. However, this volume is expected to increase to 85 dwMmtons to 110 dwMmtons by year 2020 (AIM, 2013). Table 2.2 described six types of biomass from oil palm sector that includes five types of solid biomass and one liquid by-product which is palm oil mill effluent (POME). Table 2.2 also includes the annual yield of respective biomass reported by AIM (2012) and the corrected value by Zwart (2013) according to the fact that the oil palm trees have an average life span about 25 years. Table 2.2 showed that the highest biomass yield from oil palm sector is oil palm frond with annual yield up to 47.7 dwMmtons.

Table 2.2: Main types of biomass from oil palm sector and their respective annual yield

Biomass types	Description	Site of production	Biomass yield [a]	
			by AIM (2012)	by Zwart (2013)
Oil palm fronds (OPF)	Leaves of oil palm tree	Plantation	46.4	47.7
Oil palm trunks (OPT)	Tree trunks available at end of plantation lifecycle (25years to 30 years)	Plantation	14.4	13.0
Mesocarp fibre (MF)	Remains after crude palm oil extraction from fruit bunches	Mill	6.9	7.1
Empty fruit bunch (EFB)	Remains after removal of palm fruits	Mill	6.7	6.7
Palm kernel shells (PKS)	Remains after palm kernel oil extraction	Mill	4.1	4.0
Palm oil mill effluent (POME)	Liquid by-product from sterilization and milling process of fresh fruit bunch	Mill	59.3[b]	3.0

Note: [a] in dry weight million metric tons per year
[b] in wet weight million metric tons per year

Biomass need to be optimally exploited to obtain maximum benefits for their uses. Therefore, the National Biomass Strategy (NBS) 2020 had laid the foundations for Malaysia to capitalize on its biomass by channeling it into higher value downstream uses. So far, NBS 2020 had focused on the abundant oil palm biomass but currently, NBS is in the process to extend the scope to include all types of biomass from sources such as rubber, wood and rice husk. For example, woody biomass is one of the potential lignocellulosic biomass in Malaysia, where 2.7 Mt of woody biomass is available in Sarawak alone, predominantly from the existing plantations (AIM, 2013).Woody biomass which contained high lignin content from 18 % to 35 % (Ye and Jiayang, 2002) is a potential feedstock for the biochar production and this will be discussed further in Section 2.1.3.

2.1.3 Lignocellulosic Biomass

The term ‘lignocellulosic biomass’ is used to refer higher plants, either softwood or hardwood (Harmsen et al., 2010). Lignocellulosic biomass is the most abundant renewable material in the world. The annual production of lignocellulosic biomass worldwide is estimated to be 10 billion tonnes to 50 billion tonnes and the production covered approximately 50 % of the biomass in the world (Claassen et al., 1999).

Lignocellulose biomass is a potential source of starting materials for many industrial processes. The advantage of this biomaterial is that its processing is or will shortly become less expensive than petroleum, it will not affect food supplies and all chemicals derived from it will have a lower environmental impact than petrochemicals. Additionally, it is considered carbondioxide-neutral because burning it with coal in power plants does not add carbon to the environment beyond what was required for the process of growth (Lucia, 2008).

The main polymers of lignocellulosic biomass are cellulose, hemicellulose and lignin. Table 2.3 shows the percentage of cellulose, hemicellulose and lignin in some lignocellulosic biomass. From Table 2.3, it showed that stems from the hardwood and softwood contained the highest percentage of cellulose, leaves had the highest hemicellulose content while nut shells and tapioca stems had the highest lignin content.

Table 2.3: Cellulose, hemicellulose and lignin content in common agricultural residues

Lignocellulosic biomass material	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Reference
Hardwood stems	40−45	24−40	18−25	Ye and Jiayang (2002)
Softwood stems	45−50	25−35	25−35	
Nut shells	25−30	25−30	30−40	
Corn cobs	45	35	15	
Grasses	25−40	35−50	10−30	
Wheat straw	30	50	15	
Leaves	15−20	80−85	0	
Switch grass	45	31.4	12	
Oil palm empty fruit bunch	23.73	21.55	29.15	Mohammed et al. (2011)
Oil palm shell	27.7	21.6	44	Abnisa et al. (2011)
Rice husk	37.15	23.87	12.84	Xiujuan et al. (2011)
Tapioca stem	35.2	24.3	33.8	Han et al. (2011)
Tapioca rhizome	27.82	39.67	21.71	Pattiya et al. (2006)

The different proportions cellulose, hemicellulose and lignin in different biomass influence the product distributions on biomass pyrolysis process. Figure 2.1 showed the primary products from cellulose, hemicellulose and lignin. As can be seen from the Figure 2.1, cellulose, hemicellulose and lignin had contributed to the formation of non-condensable gases and organic liquid during the pyrolysis process. Meanwhile, the solid product or biochar was mainly derived from the cellulose and lignin.

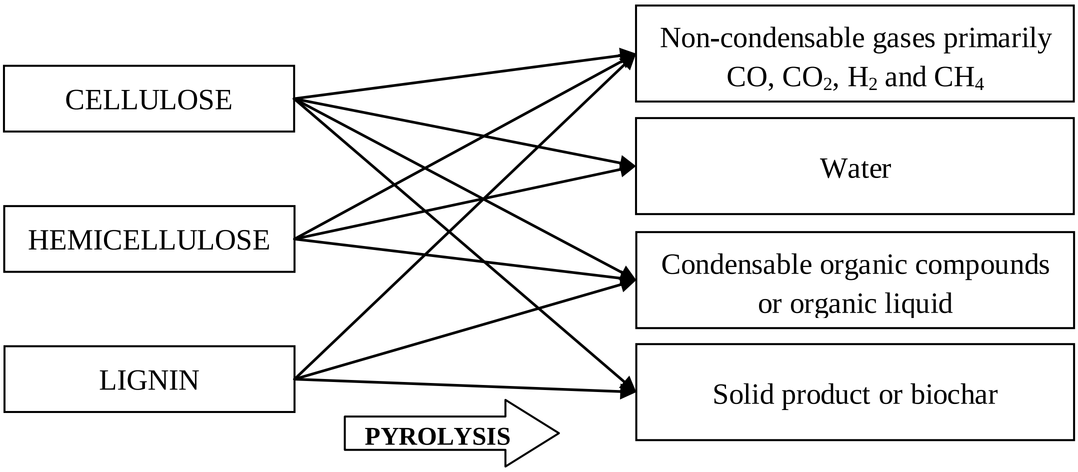
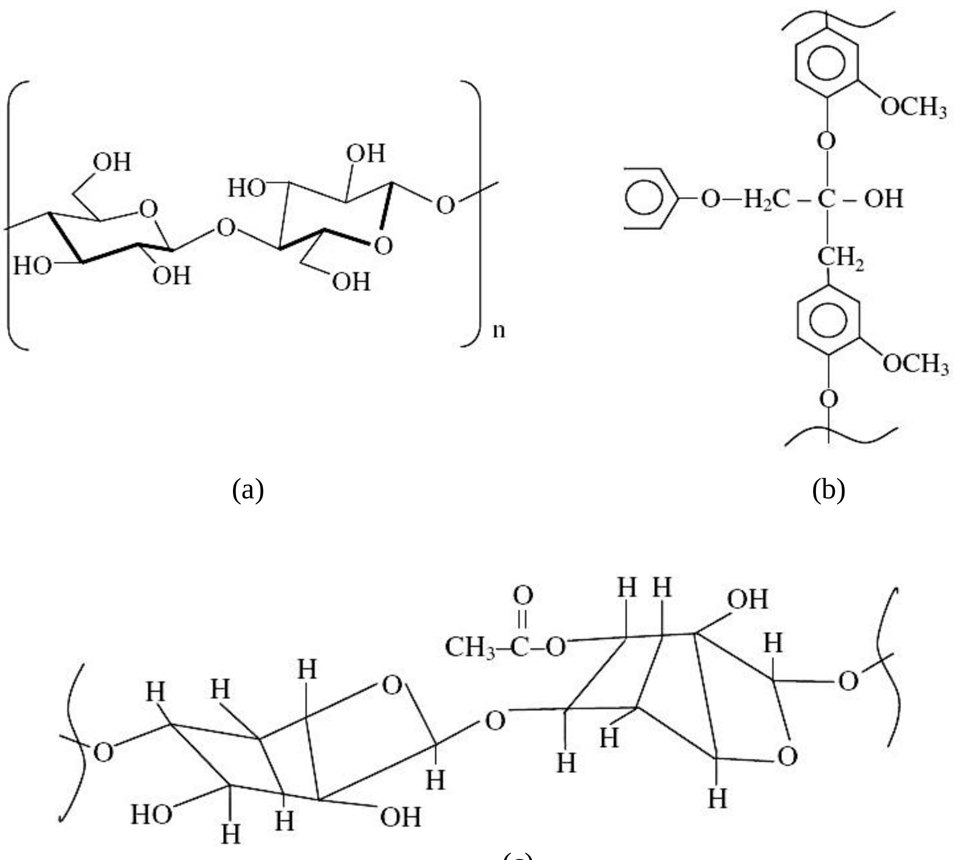


Figure 2.1: The product distribution of cellulose, hemicellulose and lignin via biomass pyrolysis process, adapted from Brown (2009)

Figure 2.2 (a), Figure 2.2 (b) and Figure 2.2 (c) respectively shows the polymers or the chemical structures for cellulose, lignin and hemicellulose. From Figure 2.2 (a), it shows that different to the hemicellulose, cellulose consisted of a long polymer of glucose without branches. The structure of cellulose is in a good order and very strong and its thermal stability is high (Yang et al., 2007). Meanwhile, Figure 2.2 (b) shows that lignin is full of aromatic rings with various branches that make it difficult to dehydrate during pyrolysis. This caused both cellulose and lignin produce more residual solid product known as biochar (Brown, 2009, Yang et al., 2007, Raveendran et al., 1996).

From the thermal degradation analysis of lignin, it shows that the activity of the chemical bonds in lignin covered an extremely wide temperature range from 100 °C to 900 °C. Lignin begins to decomposed slowly and steadily from 160 °C to 900 °C and generating high yield of solid residues or biochar to more than 45 % by weight of the original sample (Yang et al., 2007). A study by Raveendran et al. (1996) showed that the biochar yield from lignin can be up to 50 %.



(a) (b)

(c)

Figure 2.2: The chemical structures of (a) cellulose (b) lignin and (c) hemicellulose (Kabir et al., 2012)

2.1.4 Tapioca Wastes as a Potential Lignocellulosic Biomass

Nigeria, Brazil, Indonesia and Thailand are the world's largest tapioca producers. In year 2011, Nigeria is on the top with more than 52 million metric tons of tapioca production (FAO, 2011). In Malaysia, the process of tapioca planting and harvesting were conducted all year around. The tapioca production in Malaysia in year 2012 is 40,998 tons from the harvested area of 3,053 hectares, where the production increased by 23.47 % compared to year 2011 (FAO, 2014). Tapioca was planted in Malaysia mainly for the starch extraction, particularly for making monosodium glutamate that used about 3,000 tons of starch per month (Onwueme, 2002, Lian and Idris, 2000, Howeler, 2006).

Apart from its traditional role as a food crop, tapioca increased its value as a bio-fuel crop by became a major source for the ethanol bio-fuel production in many countries including Thailand, China and Africa (Kuiper et al., 2007, Onwueme, 2002, Godfrey, 2006, Jansson, 2010). The advantage of using tapioca for ethanol production is it can be grown and harvested throughout the year because of its capacity to stand low quality soils and adverse climate. This results in a constant supply of tapioca for the ethanol production, in contrast to other seasonally traditional crops such as sugarcane (Kuiper et al., 2007, Leite, 2004).

Tapioca is a crop with considerable potential for increasing productivity because it mostly grown in small plots using traditional methods, with little or non-modern inputs. Its morphological characteristics allow to take advantage of long periods of abundant rains and thus to withstand long droughts (Leite, 2004). The soils used for the tapioca planting are usually low in fertility and there is a frequent need to apply fertilizers and organic manures. In most of the countries, the soils used for tapioca plantation have high clay content, acidic with pH ranged from 4.5 to 6.5 and low in soil organic matter (Onwueme, 2002). Tapioca shall be harvested at the age of 8 months to 18 months, but the appropriate age is 12 months (TAS, 2010). Figure 2.3 illustrates the parts of a tapioca tree, with a dotted line that separates the upper ground with the underground.

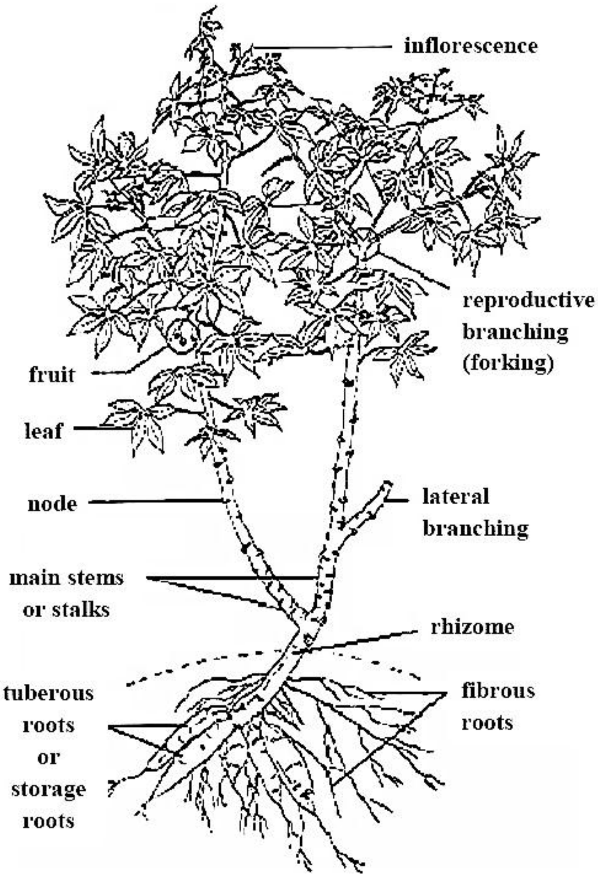


Figure 2.3: Illustration of a tapioca tree

The high demand of tapioca had increased the agriculture wastes from the tapioca plantation, for example tapioca stem and tapioca rhizome. Referring to Figure 2.3, the tapioca stem is located above the ground, while the tapioca rhizome is a part between stem and the underground tuberous roots. Table 2.4 shows the properties of tapioca stem and tapioca rhizome compared to a perennial wood known as Cassia wood.

Table 2.4: Characteristics of tapioca stem and tapioca rhizome compared to Cassia wood

Characteristics	Tapioca stem	Tapioca rhizome		Cassia wood
	Pattiya et al. (2007)	Jongpluempiti and Tangchaichit (2012)	Pattiya et al. (2007)	Jongpluempiti and Tangchaichit (2012)
Proximate analysis, (mf wt. %)				
Moisture	15.54[a]	11.49	8.31[a]	1.90
Volatile matter	79.90	76.30	77.75	76.84
Ash	6.01	1.81	4.05	2.67
Fixed carbon	14.09	10.41	18.20	18.60
Ultimate analysis, (mf wt. %)				
Carbon, C	51.12	n/a	51.59	n/a
Hydrogen, H	6.87	n/a	6.69	n/a
Nitrogen, N	0.67	n/a	1.27	n/a
Sulphur, S	< 0.01	n/a	< 0.01	n/a
Oxygen, O	41.34	n/a	40.45	n/a
Calorific value, (MJ/kg)	17.58	17.24	23.67	18.29

Note: [a] wt. % as received basis

Tapioca stem and tapioca rhizome contained high volatile matter and calorific value. By referring to Table 2.4, the volatile matter and calorific value for both tapioca wastes shows to be quite similar to the Cassia wood. Tapioca stem contained 79.90 mf wt. % of volatile matter and 17.58 MJ/kg of calorific value (Pattiya et al., 2007). Meanwhile, tapioca rhizome contained volatile matter that ranged from 76.30 mf wt. % to 77.75 mf wt. % and calorific value ranged from 17.24 MJ/kg to 23.67 MJ/kg (Pattiya et al., 2007, Jongpluempiti and Tangchaichit, 2012). These properties make both tapioca wastes as the suitable feedstock to undergo the thermochemical biomass conversion process such as pyrolysis. In addition, since both tapioca stem and tapioca rhizome contained low percentage of nitrogen and sulphur elements, which is less than 1.30 mf wt. %, these tapioca wastes will only gives off low rates of the polluting nitrogen oxides (NO_x) and sulphur oxides (SO_x) when pyrolyzed as the feedstock.

There are many studies were done on the tapioca wastes but there are no systematical researches reported about the properties of biochar derives from the tapioca wastes. For example, Pattiya (2011) used tapioca stem and tapioca rhizome as the feedstock to produce and characterize the liquid product or bio-oil derived from fast pyrolysis using a fluidized-bed reactor. The study showed that, tapioca rhizome was a promising feedstock for the bio-oil production compared to tapioca stem. The bio-oil produced from tapioca rhizome was in better quality and the yield was slightly higher with approximately 2 wt. % to 4 wt. % more than the percentage yield of bio-oil from tapioca stem.

Another study done by Pattiya et al. (2013) to investigate the effect of washing pre-treatment techniques on ash content of tapioca stem and tapioca rhizome. The results showed that the ash content in both tapioca wastes was reduced by pre-treatment such as size reduction and screening, ambient temperature water washing, hot water washing and acid washing. The most effective pre-treatment to reduce the ash content of both tapioca wastes was by acid washing that can remove up to 60 % of the original ash content.

2.2 Overview of Pyrolysis Conversion Processes

2.2.1 Introduction

Two main routes for biomass conversion process are thermochemical and biological conversion processes (McKendry, 2002, Verma et al., 2012). There are many advantages of thermochemical conversion process compared to the biological process. The comparison between this two biomass conversion processes were summarized in Table 2.5.

Table 2.5: Advantages of thermochemical conversion of biomass over the biological process (Verma et al., 2012)

Thermochemical	Biological
a) Effectively applied to almost any biomass feedstock.	a) Involves the use of microbes, enzymes and/or chemicals to utilize the limited range of biomass.
b) Relatively higher productivity (production per unit time) due to completely chemical nature of reaction.	b) Productivity is limited. Higher productivity would require higher capital investment such as bigger reactor.
c) Multiple high-value products possible using fractional separation of products.	c) Normally, limited to one or few products and would require additional microbial culture, enzymes for more products.
d) Independent of climate conditions, operates at much higher temperature range, therefore, effect of ambient temperature will be minimal.	d) Mostly susceptible to ambient temperature, and so forth such as anaerobic digester, sunlight for algal ponds.
e) Mostly complete utilization of the waste/biomass.	e) Production of secondary wastes such as biomass sludge.

Thermochemical conversion can be divided into pyrolysis, combustion, gasification and liquefaction (McKendry, 2002, Bridgwater and Peacocke, 2000, Balat, 2008). Figure 2.4 represent the product yield from each thermochemical conversion processes.

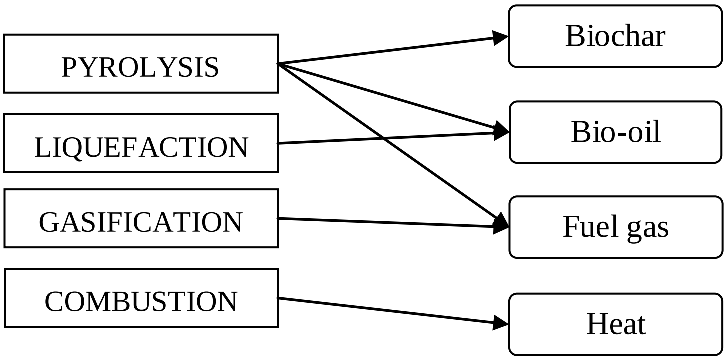


Figure 2.4: Thermochemical biomass processes and their respective products (Bridgwater and Peacocke, 2000)

Among all thermochemical conversion processes, pyrolysis plays a key role in the reaction kinetics and hence in determining product distribution, composition, and properties (Raveendran et al., 1995). Pyrolysis can be described as the thermal decomposition of biomass in the absence or restricted oxygen to obtain liquid product or bio-oil, solid product or biochar and gaseous fraction (Balat et al., 2009, Verma et al., 2012). Pyrolysis offers a great opportunity from the sustainable development point of view since it allows the use of a wide variety of materials as the feedstock and produced low emission green house gases, compared to the other technologies that are used in the process of incineration (Zajec, 2009, Verma et al., 2012).

Sinha et al. (2000) claimed that the general mechanism of pyrolysis are: heat transfer from a heat source to increase the temperature inside the fuel; initiation of primary pyrolysis reactions due to the increased temperature leading to the release of volatiles and the formation of char; flow of hot volatiles toward cooler solids results in heat transfer between hot volatiles and cooler un-pyrolyzed fuel; condensation of some of the volatiles in the cooler parts of the fuel, followed by the secondary reaction to produce tar and proceed to the auto-catalytic secondary pyrolysis. Then, according to Mohan et al. (2006), the pyrolysis process undergo further thermal decomposition, reforming, water gas shift reactions, radicals recombination and dehydrations, which are a function of the process's residence time, temperature and pressure profile.

The product stream from pyrolysis depended on the heating rate and the duration of heating process known as the residence time (Hodge, 2010). If the purpose is to maximize the yield of liquid products resulting from biomass pyrolysis, a low temperature, high heating rate and short residence time process would be required. For a high production of solid or biochar, a low temperature, low heating rate process and longer residence time would be chosen. If the purpose is to maximize the yield of fuel gas resulting from pyrolysis, a high temperature, low heating rate, long residence time process would be preferred (Demirbas, 2006). Referring to Figure 2.4, pyrolysis products for instance bio-oil and biochar can have various applications such as source of energy, chemical feedstock for industries, liquid fuels, carbon sequestration, bio-remediation, and soil enhancement. On the other hand, gasification and combustion have limited uses to heating and energy (Verma et al., 2012).

There are many researchers have studied about pyrolysis of different biomass including oil palm trunks (Deris et al., 2006), oil palm shells (Abnisa et al., 2011), oil palm pressed fruit fibres (Khor et al., 2009), oil palm empty fruit bunches (Abdullah and Gerhauser, 2008), sugarcane bagasse (Das et al., 2004), cottonseed cake (Özbay et al., 2001), black cumin seed cake (Şen and Kar, 2011), safflower seed (Beis et al., 2002), pine wood (Williams and Besler, 1996), tapioca stalk or tapioca stem and tapioca rhizome (Pattiya, 2011).

The main types of pyrolysis process are slow pyrolysis, fast pyrolysis, flash pyrolysis and hydropyrolysis which will be discuss further in Section 2.2.2, Section 2.2.3, Section 2.2.4 and Section 2.2.5 respectively.

2.2.2 Slow Pyrolysis

Slow pyrolysis involved a process where feedstock is heated at a low heating rate in the range from 5 °C/min to 30 °C/min (Downie et al., 2009) or less than 50 °C/min (Patwardhan, 2010) with restricted air flow (Wereko-Brobby and Hagen, 1996) or in the absence of oxygen (Basu, 2010, Goyal et al., 2008). The terminal temperature for slow pyrolysis is in the range from 400 °C to 600 °C (Basu, 2010) while the residence time varies between 30 minutes to several hours (McCarl et al., 2009).

Size of biomass feedstock is prepared in different ways, depending on the heating rate of a pyrolysis process. For the pyrolysis that requires faster heating rate, smaller feedstock particles are needed to facilitate the heat and mass transfer of the pyrolysis reactions (Downie et al., 2009). Meanwhile, for slow pyrolysis which employs slower heating rates and longer residence time, this process can accommodate feedstock with larger size up to several centimeters in dimension.

Slow pyrolysis yields relatively more biochar. The distribution of products from slow pyrolysis is approximately 35 % of biochar, 30 % of bio-oil and other by-products (Ringer et al., 2006). Current developments and researches on slow pyrolysis process are having most interest for the biochar production because there are various advantages of biochar when used as a soil amendment. This subject has been covered extensively by Lehmann and Joseph (2009) and in other research literature. In this thesis, Section 2.3.2 discussed about the biochar application as the soil amendment, while Section 2.3.3 reported about the biochar production from different parameters.

2.2.3 Fast Pyrolysis

Fast pyrolysis is a process in which the feedstock is rapidly heated in the absence of air, vaporizes and condenses to a dark brown mobile liquid known as the bio-oil. According to Bridgwater and Peacocke (2000), heating value of bio-oil is about half of the heating value of conventional fuel oil.

Since fast pyrolysis process involved high heating rate and high heat transfer, it usually requires a finely ground feedstock (Hodge, 2010, Bridgwater and Peacocke, 2000, Bridgwater et al., 1999). For example, Pattiya (2011) had studied about fast pyrolysis of tapioca stem and tapioca rhizome. Both feedstock were grounded to particle sizes ranged from 355 µm to 500 µm. Meanwhile, Abdullah and Bridgwater (2006) used the feedstock of oil palm empty fruit bunch with sizes ranged from 250 μm to 355 μm to produced bio-oil via fast pyrolysis using a 150 g/h fluidized bed reactor.

During fast pyrolysis, feedstock was heated with a heating rate that can be as high as 1,000 °C/s to 10,000 °C/s and residence time of less than 2 seconds (Bridgwater et al., 1999) or even less than 0.5 seconds (Hodge, 2010). The generated vapor was cooled rapidly then condensed to produce the main product, bio-oil (Bridgwater and Peacocke, 2000). Rapid cooling or rapid quenching process is necessary during fast pyrolysis to prevent the high molecular weight liquids from decomposed into gases with lower molecular weight (Hodge, 2010).

The percentage yield of bio-oil from fast pyrolysis can be optimized at pyrolysis temperatures ranged from 400 °C to 600 °C (Basu, 2010, Hodge, 2010). According to Bridgwater and Peacocke (2000), for fast pyrolysis of wood, maximum bio-oil yield up to 80 wt. % on dry basis can be obtained at a pyrolysis temperature of 500 °C.

2.2.4 Flash Pyrolysis

Flash pyrolysis is a type of pyrolysis characterized by a very high heating rate of more than 1,000 °C/s and short residence times which is less than 1 second. Flash pyrolysis involves different temperature range depending on the wanted product. Table 2.6 indicates the operating parameters and the main products of two types of flash pyrolysis processes known as flash-liquid and flash-gas. For flash-liquid, the pyrolysis temperature is in the range from 450 °C to 750 °C and the main product is liquid fraction or bio-oil that yield up to 80 wt. %. Meanwhile, flash-gas pyrolysis involved high speed reaction at temperature exceeding 750 °C to produce gas as the main product that yield up to 80 wt. % (Zajec, 2009).

Table 2.6: Types of flash pyrolysis in relation to operating parameters and its main product (Zajec, 2009)

Type of flash pyrolysis	Residence time	Temperature	Heating rate	Main product
Flash-liquid	< 1s	450 °C–750 °C	> 1000 °C/s	Bio-oil
Flash-gas	< 1s	> 750 °C	> 1000 °C/s	Gas

According to Balat (2008) the conversion of biomass to bio-oil via flash pyrolysis can have high efficiency of up to 70 %. However, Brem and Bramer (2007) stated that the major problem of flash pyrolysis is the quality and the stability of the produced bio-oil was strongly affected by the char or the ash content. Fine chars in the bio-oil catalyzed the repolymerization reactions resulting in higher viscosity oil, which is disadvantageous. The char can be removed after treatment of condensed products for example by filtering the bio-oil.

2.2.5 Hydropyrolysis

Normal pyrolysis, for example slow pyrolysis, fast pyrolysis and flash pyrolysis were generally carried out in the absence of a medium such as air. However, some special types of pyrolysis technology were conducted in a specific medium, for instance hydropyrolysis that took place in the medium of high-pressure H_2. Hydropyrolysis is a thermal decomposition of biomass at high heating rate, mainly for the production of bio-oil. The residence time of hydropyrolysis is less than 10 seconds at temperatures less than 500 °C (Basu, 2010).

Hydropyrolysis can increase the volatile yield and the proportion of lower molar mass hydrocarbons (Rocha et al., 1997). The high volatile yield is due to the free-radical fragments that are sufficient to stabilize them before they repolymerize and form biochar (Basu, 2010). Consequently, liquid product known as bio-oil is the main product yield from the hydropyrolysis process. Meier et al. (1995) have conducted hydropyrolysis experiments using various feedstock of biomass and technical lignins. The results showed that the conversions of all feedstock were excellent and generally the liquid yields were very high up to 80 %.

High oxygen content is an important shortcoming of bio-oil that makes them inferior to the conventional fuels. Hydropyrolysis is a process that can produce bio-oil with reduced oxygen (Basu, 2010). Rocha et al. (1999) had perform the fixed bed hydropyrolysis experiments on cellulose, sugar cane bagasse and eucalyptus. The results from nuclear magnetic resonance (NMR) spectroscopy indicated that bio-oil produced at higher

H_2 pressure became increasingly aromatic as more oxygen was removed. H_2 pressures up to 10 megapascals had reduced the oxygen content of bio-oil by 10 wt. % to 20 wt. %.

2.3 Overview of Biochar

2.3.1 Introduction

Biochar production and utilization systems are carbon negative technology where it removes the net CO_2 from the atmosphere and stores it as the stable soil carbon sinks (Lehmann et al., 2006). Biochar can be used as the soil amendment for the carbon sequestration.

2.3.2 Biochar as the Soil Amendment

Interest towards the impacts of biochar on soils and plant growth was first stimulated by the remarkable discovery of ‘terra preta’ soils in Amazonia. ‘Terra preta’ was a soil created by the pre-historic Amazonian that applied high amount of charred vegetable, charred animal matter, pottery and other midden-waste to their wet desert soil known as ‘oxisol’. ‘Terra preta’ soil was more fertile than other tropical soils and it sustained without much nutrient having leached out even after 2,000 years (APN, 2009, Antal and Grønli, 2003).

Lehmann (2007) reported, two main aspects of biochar that make it so valuable as a soil application compared to other soil organic matter (SOM) are its high stability against decay and superior ability to retain nutrients. These two aspects are the key advantages of biochar with respect to the soil ecosystem functions. Besides, International Biochar Initiative (IBI) also found to agree when stated that biochar tends to hold on to nutrients and likely helps to reduce their loss by leaching, so the nutrients remain available to use by plants. Besides, many biochar also contained ash which can be served as a fertilizer.

A report for a project by Asia-Pacific Network for Global Change Research (APN, 2009) summarized some possible reasons which help to account for the positive impacts of biochar on soils. The reasons are:

- Increased in pH of acidic soils since biochar was typically alkaline;
- Increased in water retention, especially in sandy and silty soils;
- Provision of nutrients in the ash contained within the biochar;
- Enhancement of the cation exchange capacity (CEC) of soil that increased the efficiency of nutrient use;
- Enhancement of microbial communities within biochar, included bacteria, mycorrhizae and fungal hyphae, with knock-on benefits to soil processes;
- Benefits to the physical structure of soil for example through increased in porosity of soil to water incident at the surface.

There are many authors had reported that the application of biochar on farm fields increased the yield of agricultural crops. Table 2.9 represent the summary of increased of crop yield, including bean, maize and rice and the amount of biochar added to the respective agricultural soils.

Table 2.9: The increased of crops yield from the field trials with biochar

Crop	Amount of biochar added to agricultural soils	Increased of crop yield	Reference
Bean	90 g/kg	46 %	Rondon et al. (2007)
Maize	20 t/ha	28 %	Rondon et al. (2006)
Rice	11 Mg/ha	> 100 %	Nehls (2002)

An experiment done by Kwapinski et al. (2010) showed that the maize plant had grew better using the soil mixed with higher amount of biochar, regardless the types of biochar. In the study, three types of biochars were prepared from different feedstock of willow chips, pine chips and miscanthus chips at pyrolysis temperature of 500 °C for 10 minutes. Each of the biochars produced were mixed with a clay loam soil at rates of 1 wt. % biochar and 5 wt. % biochar and added to the maize seeds in the pots. No biochar was added to the control pot. Six maize seeds were planted per pot and incubated in the greenhouse for 21 days. The results showed that the percentage of growth for plants grown