UNIVERSITI PUTRA MALAYSIA

BIOCHAR PRODUCTION FROM SAGO (Metroxylon Spp.) VIA PYROLYSIS

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By

JAKARIA BIN RAMBLI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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DEDICATION

I would like to dedicate this thesis to my parents for their endless love, support and encouragement. Thank you both for giving me strength to reach for the starts and chase my dreams.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

BIOCHAR PRODUCTION FROM SAGO (*Metroxylon* Spp.) VIA PYROLYSIS

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Chairman : Assoc. Prof Wan Azlina Binti Wan Abdul Karim Ghani, PhD, Ir
Faculty : Engineering

The limited of fossil fuels and the growing awareness of the detrimental environmental consequences resulting from greenhouse gas emissions have reinforced the importance of biomass as an energy resource in developed and developing countries. It is expected that future energy use will have increased utilization of different energy sources, including biomass, municipal solid wastes, industrial wastes, agricultural wastes and other low grade fuels. Recently, the ease of accessibility of sago biomass has drawn considerable interest of researches regarding the production of renewable energy. Pyrolysis is a good practical solution to solve the growing problem of landfills, with simultaneous energy extraction and nonleachable minimum residue. Pyrolysis also provides good solution to the problem of sago residue particularly in the region of Sarawak, Malaysia. Therefore, an effort is made in this study to utilize sago biomass as agricultural residue for the production of cost effective and environmental friendly fuel. Furthermore, the slow pyrolysis of sago biomass from different sources of the plant (bark, frond and cortex) by using Electrical Furnace Reactor was studied with the aim of producing solid pyrolysis product known as biochar, having promising properties and potential for use in traditional fossil coal applications. The study focuses on investigating of the role of best process parameters including reaction temperature, process time and nitrogen flow rate on production of biochar. The experiments were designed using central composite design (CCD) method and the optimization was performed by using response surface methodology. The characteristics of biochar based on its quality, distribution of chemical species, carbon conversion efficiency and thermal efficiency has been examined. Optimal conditions was obtained at the temperature of 400 ºC, 20 minutes of process time and nitrogen flow rate of 75 mL/min to result in the maximum yield of biochar at 47%. Moreover, the calorific value was remarkably improved from 22.16 MJ/kg to 25.92 MJ/kg as the biomass was turned into biochar. The locally sourced starch flours were utilized as binder to produce three different grades of briquettes from the produced biochar at different mixing ratios. The textural, morphological and thermal stability characteristics of the prepared briquettes were investigated by surface area analysis (Brunauer-Emmett-Teller equation), scanning electron microscopy (SEM), and Thermogravimetric analysis (TGA). The sago cortex was found to have lower ash content as compared to other types whereas the mean calorific value of the briquettes were found to be 21.63 MJ/kg, 23.23 MJ/kg and 22.33 MJ/kg for sago barks, sago fronds and sago cortex, respectively. Experimental results showed that sago biomass is a potential alternative fuel for current fuels.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

Biochar Yang Dihasilkan Daripada Sago *(Metroxylon* Spp.*)* Melalui Pirolisis

Oleh

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Pengerusi : Prof. Madya Wan Azlina Binti Wan Abdul Karim Ghani, PhD, Ir
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Tenaga dianggap asas bagi kemajuan dan kemakmuran negara dan masyarakat. Ia juga merupakan asas pembangunan ekonomi dan sosial. Akibat daripada bahan api fosil yang semakin menurun menyebabkan peningkatan kesedaran terhadap kesan-kesan alam sekitar yang memberi impak negatif akibat pelepasan gas rumah hijau telah membuktikan kepentingan biomas sebagai sumber tenaga di negara maju dan membangun. Baru-baru ini, kemudahan akses biogas sago telah menarik minat penyelidikan mengenai pengeluaran tenaga boleh diperbaharui. Dijangkakan penggunaan tenaga diperbaharui masa hadapan akan meningkatkan pemanfaatan sumber tenaga yang berbeza, termasuk biomas, sisa pepejal perbandaran, sisa industri, sisa pertanian dan bahan api kelas rendah yang lain. Dengan kaedah pyrolysis perlahan menggunakan pelbagai jenis biomas sago (isi, pelepah dan kulit) dikaji dengan tujuan menghasilkan produk pirolisis padat (biochar) dengan ciri-ciri yang menjanjikan dan berpotensi digunakan dalam aplikasi arang batu fosil tradisional. Pyrolysis adalah penyelesaian praktikal yang baik bertujuan menyelesaikan masalah pertumbuhan tapak pelupusan, dengan pengekstrakan tenaga serentak dan sisa minimum yang tidak dapat dilepaskan. Pyrolysis juga menyediakan penyelesaian yang baik kepada masalah sisa buangan sagu kepada bahan api yang berguna. Dalam kajian ini, usaha dibuat untuk menggunakan sisa pertanian untuk pengeluaran bahan bakar mesra alam sekitar dan kos yang minima. Dengan pengetahuan yang terbaik, tidak ada penyelidikan mengenai keupayaan sisa biomas sago dalam menjana kuasa di Sarawak, Malaysia. Eksperimen pirolisis batch dilakukan di reaktor relau elektrik. Kajian ini juga memberi tumpuan kepada peranan beberapa proses parameter terbaik termasuk tindak balas suhu, masa proses dan kadar aliran nitrogen dioptimumkan menggunakan *"central composite design of response surface methodology".* Kesan tindak balas suhu, masa proses dan aliran gas nitrogen dari pelbagai jenis sampel sagu diperiksa dan dianalisa. Ciri-ciri biochar berdasarkan kualiti, spesies kimia, kecekapan penukaran karbon, kecekapan terma dan kepekatan hidrogen telah diperiksa. Keputusan membuktikan bahawa keadaan optimum untuk pengeluaran biochar adalah pada suhu tertinggi 400 ºC, 20 minit masa proses dan kadar aliran nitrogen 75 mL / min, menghasilkan jumlah maksimum Sago-Derived Biochar (SDB) iaitu sebanyak 47%. Nilai kalori SDB yang dioptimumkan telah meningkat dengan ketara dari 22.16 MJ / kg untuk biomas sago kepada 25.92 MJ / kg. Sisa biomas boleh dengan mudah ditukarkan kepada briket yang berfungsi sebagai pengganti minyak bahan api semasa. Dalam kajian ini, bahan bakar biomas yang sangat berpotensi dihasilkan menggunakan biomass sagu. Dalam hal ini, tepung kanji yang berasal dari tempatan digunakan sebagai bahan pengikat untuk menghasilkan tiga gred arang briket yang berbeza pada 5 hingga 20%. Ciri-ciri kestabilan tekstur, morfologi dan terma briket yang disediakan dinilai menggunakan analisis kawasan permukaan (persamaan Brunauer-Emmett-Teller), pengimbasan mikroskop elektron (SEM), dan analisis Thermogravimetric (TGA). Selain itu, kandungan bahan yang tidak menentu, kandungan karbon tetap dan kandungan abu telah diperiksa. Kandungan abu masing-masing ialah 24.15%, 24.15% dan 22.33% dengan ketumpatan pukal 0.630g / m3, 0.725g / m3 dan 0.620g / m3. Di samping itu, kulit sago didapati mempunyai kandungan abu yang lebih rendah berbanding dengan isi sagu dan pelepah sagu. Nilai purata kalori briket didapati 21.63 MJ / kg, 23.23 MJ / kg dan 22.33 MJ / kg untuk isi sago, pelepah sago dan kulit sago. Keputusan eksperimen menunjukkan bahawa biomas sago adalah bahan bakar alternatif yang berpotensi untuk bahan bakar semasa.

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Alhamdulillah.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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This is to confirm that:

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LIST OF ABBREVIATIONS

UPM	Universiti Putra Malaysia
A	Surface area of reactor
Al	Aluminium
Al_2O_3	Aluminium oxide
AC_{stq}	Stoichiometric air/fuel ratio
A_i	Peak area (Standard)
A_s	Peak area (Sample)
BFB	Bubbling fluidized bed
Br	Bromide
Ca	Calcium
CaO	Calcium Oxide
Cl	Chloride
CuO	Copper (II) oxide
CO	Carbon monoxide
CO_2	Carbon dioxde
NOx	Nitrogen Oxides
SOx	Sulfur Oxides
CCE	Carbon conversion efficiency
CFB	Circulating fluidized bed
CCD	Central Composite Design
CD	Drag coefficient
cp	Specific heat capacity, KJ/kg K
CV	Calorific value
CHNS	Carbon-hydrogen-nitrogen-sulpur analyzer
CH_4	Methane
db	Dry basis
dp	Particle size
R^2	R-squared
DFB	Dual fluidised bed
E	Activation energy, J/mol
EFB	Empty fruit bunch
ER	Equivalence ratio
Fe	Iron
Fe_2O_3	Iron (II) oxide
GC	Gas Chromatography
GCMS	Gas chromatography-mass spectrometry
GHG	Greenhouse gas
g	Accelaration due to gravity
h	Stoichiometric coefficient of oxgen
HHV	Higher heating value, KJ/kg or MJ/kg
He	Helium
H_2	Hydrogen
H_2O	Steam
K	Potassium
KJ	Kilo Joles
K_2O	Potassium oxide
LHV	Lower heating value, KJ/kg or MJ/kg

M	Molecular weight, kg/kmol
MSW	Municipal solid waste
Mg	Magnesium
MnO	Manganese (II) oxide
MgO	Magnesium oxide
MWe	Megawatt electrical
n	Molar flow rate, kmol/s
Na	Sodium
O_2	Oxygen
OFAT	One factor at a time
P	Phosphorus
PJ	Peta Joules
MJ	Megajoule
GJ/t	Gigajoule
P	Pressure, atm
Q	Gas volumetric flow rate
RSM	Response surface methodology
S	Sulphur
Si	Silica
T	Temperature, °C or K
TGA	Thermogravimetric analyzer
V	Volumetric flow rate
Wt.%	Weight percent
SDB	Sago-derived biochar
OB	Optimize biochar
MF	Mesocarp fiber
Wt.	Weight
Mf%	Mass fraction percent
K	Kelvin (temperature)
pJ	Picojoule
Min^{-1}	Minute per second
SB	Sago Biochar
CS	Corn Starch
TS	Tapioca Starch
NF	Nitrogen Flow
PT	Process Time
ha	hectare
Kg/m^3	Kilogram per cubic metre
°C/min	Heating rate
mt	Metric tonnes
MW	Megawatt
BET	Brunauer-Emmett-Teller
SEM	Scanning electron microscopy
CHNS	Carbon-hydrogen-nitrogen-sulphur
FTIR	Fourier transform infrared spectroscopy
XRD	X-ray powder diffractometers
GC	Gas chromatography
MPa	Megapascal
PPM	Parts per million
DOE	Design of experiment

CHAPTER 1

INTRODUCTION

1.1.1 Background

Sago plantation in Mukah division is greatly contribute to the increase of the agricultural industry in Sarawak, Malaysia. Previously, sago palms were only being used as feeds for livestock and also for traditional food production. Due to economic development, the use of sago palms were broaden and the demand of their use in other various applications were become higher. Nowadays, Sarawak is the world's largest exporters of sago supplies whereas tens of tonnes of sago are annually exported to Peninsular, Singapore, Taiwan and Japan and other countries in the region. Table 1.1 presents the amount of annual sago production in Sarawak, Malaysia follows by the prices and export values of production as reported by the Department of Agriculture (MANRED, 2018). Significant increase can be observed as comparing export value of sago between the years of 2010 to 2016. The sago export value of 45,000 tonnes (RM1500/tonnes) contributing to the value of RM60,000,000 in year 2010 whereas the export value of sago that amounted to 41,000 tonnes (RM2158/tonnes) contributed to the revenue value of RM 90,000,000 in year 2016. The total export earnings of sago showed an increase from RM 60,000,000 in year to RM 90, 000,000 in year 2016.

Table 1.1: Summarizes the values related to sago palm production in years 2010 to 2016

Sago palm	Year						
	2010	**2011**	**2012**	**2013**	**2014**	**2015**	**2016**
Quantity[a]	45,000	53,000	48,000	49,000	47,000	41,500	41,000
Price[b]	1,500	1,800	1,700	1,600	1,500	2,200	2,158
Export Value[c]	60	90	90	74	74.5	95	90

tonnes[a]; mt[b]; RM/M[c]

Source: (MANRED, 2018)

In Sarawak, the biggest state of Malaysia state, 2.64 million hectares of the total areas (12.33 million hectares) are only committed for agricultural activities. Sarawak commercial crop is mainly dominated by sago plantation in the land where 78.9% of the whole agricultural areas in 2015 were seen in form the Mukah division. Coconut, rubber, paddy and oil palm account for 0.2%, 4%, 8% and 8.9% of the areas of agriculture, respectively (MANRED, 2018). The official Sarawak government has recently taken appropriate measure to flourish the central region economics of Sarawak by establishing the Sarawak Corridor of Renewable Energy (SCORE), Malaysia. This scenario is done by enhancing ten industries which have high impact on the economy of the Sarawak.

Presently, there is a risk in the current coal fired systems for power generating using the combustion or co-combustion of biomass. It should be noted that using biomass energy offers a low-cost approach that considerably reduces releasing of carbon dioxide (CO_2) into the atmosphere (Dai et al., 2008). The increasing amount of biomass (forestry and agricultural) wastes has compelled researches to transform them into briquettes as a promising substitute for current fuels. Therefore, briquettes not only mitigate environmental pollution caused by sago wastes but also improve the production of a renewable energy (Thabuot, Pagketanang, Panyacharoen, Mongkut, & Wongwicha, 2015a). Furthermore, these agricultural and forestry biochar residues can be utilized as a stock for cooking and coal power plants (Kung & Chang, 2015; Lehmann, Gaunt, & Rondon, 2006).

From agricultural perspective, using biochar will improve soil structure, and reduce the use of herbicides in the soil due to excellent physicochemical characteristics and high porosity. Subsequently, it helps in the production of clean carbon (i.e. with less negative impact on environment in terms of emission and disposal) from the environment up to 20 percent (Thomazini et al., 2015) as well as reduction of greenhouse gas (GHG) emissions 12%-84% (Lehmann & Joseph, 2009; Meyer, Glaser, & Quicker, 2011; Thomazini et al., 2015). Hence, the use of biochar will significantly lessen ecological damages and at the same time enhance economic competitiveness as compared to the coal products.

1.2 Problem statement

Illegal disposal of biomass wastes into the jungles or the rivers is currently become a major problem to the government and environment caused by the production of agricultural waste. Significant amount of waste is generated from the industries associated with sago palm whereby the effluents are commonly disposed into the river or via open burning. Disposal of waste to the rivers brings harmful effect to the aquatic life. Moreover, the reserve land for agricultural waste disposal (landfilling) are limited in the area. All related issues are expected to diminish once with the transformation of waste into value added products. In this study, sago biomass samples are employed in thermal conversion through pyrolysis and turned into solid biochar which can be later used in preparation of briquettes. Once the high quality of briquettes are maintained, they can be used as solid fuel for energy production. As a result, this action would increase the variance of the abundance of energy resources for energy sectors in Sarawak, Malaysia. To the best of our knowledge, there is no significant research carried out on the capabilities of sago biomass in form of briquette as solid fuel to reduce the environmental concerns of inappropriate disposal of agricultural residues such as open burning or illegal dumping to the environment. Sago derived biochar with higher calorific value and reduced volume as compared to raw sago biomass, is easier to handle and storage (especially while in form of briquette), having less toxics and heavy metal compounds and is more enduring. If adequate method is chosen to transfer the biochar into briquettes, it could even further improved in terms of higher combustion efficiency and less emission of toxic and greenhouse gas to the environment.

1.3 Research Objectives

The main objectives of this study are:

a. To assess the properties of Sago *(Metroxylon Spp)* as potential fuel for biochar production through characterization analysis (CHNS, FTIR, BET, SEM, GC, GCMS and XRD).
b. To evaluate and optimize the effect of parameters on biochar production from Sago through design of experiment (OFAT and RSM).
c. To evaluate the suitability of sago biochar in form of briquette as a solid fuel in terms of consistency, mechanical strength and combustion efficiency.

1.4 Scope of Research

The evaluation of sago biomass properties is investigated through different characterization methods including Carbon-hydrogen-nitrogen-sulphur analyzer (CHNS), thermogravimetric analysis (TGA), scanning electron microscopy (SEM) and calorific value analysis. This study focuses on optimizing the yield of biochar from sago biomass with emphasis on investigating the effects of operating variables including temperature, process time and nitrogen gas flow on slow pyrolysis performance. Batch pyrolysis for biochar production using Sago were conducted in an electrical furnace reactor.

The optimized biochar underwent characterization analysis using the similar techniques used for biomass samples. The by-products of bio-oil, ash and syngas, however, were involved in different analysis of Gas chromatography-mass spectrometry (GCMS), Fourier transform infrared spectroscopy (FTIR), X-ray Powder Diffractometers (XRD) and Gas chromatography (GC). Biochar in further step of the study, was converted to briquette by using different ratio of binders (non-edible corn and tapioca starches) and underwent several methods of analysis to ensure the consistent burning, mechanically strength structure and with low emission level of toxic substances. Regarded techniques are including compressive strength test, Brunauer-Emmett-Teller (BET), observation and real-time gas analysing. At the final stage of the study, economic analysis was performed to estimate the total cost of capitals, operating and maintenance for the large-scale production of biochar from sago biomass.

1.5 Thesis Layout

This thesis consists of 5 chapters as described below:

1. Chapter 1 consists of the introduction of the undertaking research. The chapter presents the fundamental and background information which consists of the introduction, objectives, scope of research and thesis layout.
2. Chapter 2 describes the literature review which provides important theory and findings from preceding researches that is significant to the project.
3. Chapter 3 consists the methodology of the project which consists of experimental method, samples characterization and background of sago as potential as solid fuel.
4. Chapter 4 mainly justifies the results and findings of the experimental work. The results obtained will be linked to the previous theory and new discovery will be highlighted and justified.
5. Chapter 5 covers the final part of the report which summarizes the research findings and recommendation for future works.

With this preliminary study on various types of biomass and biochar, it is hoped that the discovery will be developed further and used by others as an efficient the capabilities of biomass waste in generating power in Sarawak.

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LIST OF PUBLICATIONS

Rambli, J., Ghani, W. A. W. A. K., & Salleh, M. A. M. (2018). Characterization of Sago-based Biochar as Potential Feedstock for Solid Fuel. *Journal of Energy and Safety Technology (JEST)*, *1*(2).

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An Outlook of Malaysian Biomass Industry Commercialisation: Perspectives and Challenges (Peer-Reviewed Articles in Renewable & Sustainable Energy Reviews Journal).

Sago-Based Biochar Production and its Characteristics and Potential Application Solid Fuel Feedstock (2018 International Conference on the Biomass-Environment-Food-Energy-Water (BEFEW) Nexus).

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