SLOW PYROLYSIS OF *IMPERATA CYLINDRICA* IN A FIXED BED REACTOR

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To my beloved husband, mother and father

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ABSTRACT

Slow pyrolysis of *Imperata Cylindrica* has been conducted in a fixed bed reactor to determine the effect of temperature and particle sizes towards the product yield. The characterization of the *Imperata Cylindrica* has been analysed using several instruments such as Thermogravimetric Analysis (TGA), Carbon Hidrogen Nitrogen Sulfur and Oxygen (CHNS/O) analyzer, Bomb Calorimeter, and several analytical methods. Pyrolysis experiments were performed at temperature between 450- 600 °C, and particle sizes of 0.25-1.00 mm with constant nitrogen flow rate of 100 cm^3min^{-1} and heating rate of 22 ºC . min^{-1} (slow mode). The highest liquid oil yield obtained was 20.88 % at temperature 500 °C, with particle size of 0.5-1.0 mm, and heating rate of 22 ºC . min^{-1}. The obtained yield of liquid, solid and gas from pyrolysis were found in the range of 3.25-20.88 %, 22.63-30.50 % and 49.13-74.13 % respectively at different pyrolysis conditions. Liquid bio-oil produced from the pyrolysis of *Imperata Cylindrica* shows high water content in the range of 58.09-72.74 % which was checked using Karl Fisher Titration. From Gas Chromatography-Mass Spectrometry (GC-MS), the chemical components present in the liquid oil from pyrolysis of *Imperata cylindrica* include acids, phenols, ketones, aldehydes, ethers, and some species of aromatics.

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ABSTRAK

Pirolisis perlahan *Imperata cylindrica* telah dijalankan di dalam reaktor katil tetap untuk menentukan kesan suhu dan saiz zarah terhadap hasil produk. Pencirian cylindrica Imperata telah dianalisis dengan menggunakan beberapa instrumen seperti Termogravimetri Analisis (TGA),Penganalisa Sulfur Nitrogen Karbon Hidrogen dan Oksigen (CHNS/O), Bom Kalorimeter, dan beberapa kaedah analisis. Eksperimen pirolisis telah dijalankan pada suhu antara 450-600 ° C, dan saiz zarah 0.25-1.00 mm dengan kadar aliran nitrogen malar 100 cm^3. min^{-1} dan kadar pemanasan 22 º C . min^{-1} (mod perlahan). Hasil minyak cecair tertinggi yang diperolehi adalah 20.88 % pada suhu 500 °C, dengan saiz zarah 0.5-1.0 mm, dan kadar pemanasan 22 º C . min^{-1}. Hasil yang diperolehi cecair, pepejal dan gas daripada pirolisis ditemui dalam julat 3.25-20.88 %, 22.63-30.50 % dan 49.13-74.13 % masing-masing pada keadaan pirolisis yang berbeza. Cecair bio-minyak yang dihasilkan daripada pirolisis *Imperata cylindrica* menunjukkan kandungan air yang tinggi dalam julat 58.09-72.74 % yang telah diperiksa menggunakan Pentitratan Karl Fisher. Daripada Gas Kromatografi-Jisim Spektrometri (GC-MS), komponen kimia yang hadir dalam minyak cecair daripada pirolisis Imperata cylindrica termasuk asid, fenol, keton, aldehid, eter, dan beberapa spesies aromatik.

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LIST OF ABBREVIATIONS

VM	-	Volatile Matter
FC	-	Fixed Carbon
Ad	-	Air dried
TGA	-	Thermogravimetric Analysis
GC-MS	-	Gas Chromotography Mass Spectrometry
CHNS/O	-	Carbon, Hydrogen, Nitrogen, Sulfur, Oxygen
HHV	-	High heating Value
LHV	-	Low Heating value
UTM	-	University Technology Malaysia

LIST OF SYMBOLS

ºC	-	degree celcius
wt. %	-	weight percent
D	-	Diameter
T	-	Temperature
g	-	gram
min	-	min
mm	-	milimeter
t	-	time

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CHAPTER 1

INTRODUCTION

1.1 General Background

Nowadays, biomass has widely acknowledged as a source of alternative energies, which are more sustainable due to its great potential compared to fossil fuel in satisfying environmental aspects. And the most important, these kinds of alternative energy providing the only supply of renewable solid, liquid and gaseous fuels (Bridgwater, 2002).

Recently, there have been lots of studies in the field of thermo chemical conversion of biomass into liquid, gaseous or solid products. There are three types of thermal conversion technologies, which are combustion, gasification, and pyrolysis. Among all the process, pyrolysis is still at premature stage of improvement. Pyrolysis can be categorized in two modes process, which are slow pyrolysis and fast pyrolysis, which depends on the operational and also the main product where for fast pyrolysis the main product is bio-oil, and for slow pyrolysis the main product is char (Bridgwater, 1997).

In general, all types of biomass can be thermo chemically converted into bio-oil. In this work, *Imperata cylindrica* or also known as '*lalang*' in Malaysia*,* were chosen as biomass feedstock since they are available in large quantity throughout the tropical and subtropical regions of the world. The bio-oil or liquid fuel produced from the pyrolysis process has wide application as a fuel and also for the production of various types of chemicals.

1.2 Problem Statement

Slow pyrolysis is often related to the production of charcoal while fast pyrolysis was related with production of bio-oil. Bridgwater (2003) have shown that slow pyrolysis of biomass will produce high content of charcoal. Researches in the world are more focusing on fast pyrolysis since it was the promising routes in producing bio-oil and this have cause neglecting of conventional (slow) pyrolysis process although slow pyrolysis also permits in producing solid, liquid, and gaseous yields in considerable portions (Demirbas, 2004).

The product from pyrolysis is highly dependent on the feedstock and the process conditions employed the pyrolysed liquid which known as bio-oil and it can yields up to 70–75 wt% from wood (Oasmaa et al., 2005). However, bio-oils are still considered as low quality liquid fuels compared to petroleum based fuel, due to its poor properties such as complex multiphase structures, low heating values, high contents of water, oxygen, solids and ash, high surface tension and viscosity, have instable chemical and thermal, pH values is low (acidic) , and also poor ignition and combustion properties. Although bio-oils are lacking in some properties compared to petroleum fuel, it does have some specialty in lubricate properties. Furthermore, bio-oils are more environmental friendly since they are biodegradable and less toxic as well. The challenge of research today is to develop bio-oils as commercialized liquid fuels to compete with existing petroleum fuels.

Some factors might affect the chemical composition of bio-oil such as biomass feedstock type, pyrolysis parameters (residence time, pressure, temperature, gaseous environment heating rate), pre-treatment process, and also condensation and vapour filtration (condensing method and medium, filter type, cooling rate). Hence, the different use of biomass feedstock and different reactor configuration will give different results among them. As a result, the fuel properties of different bio-oils usually vary in wide ranges.

1.3 Objectives

The objectives of this research are:

i) To characterize physical and chemical properties of *Imperata cylindrica* as a biomass feedstock for pyrolysis.

ii) To design and set up the rig for slow pyrolysis process.

iii) To study the effect of pyrolysis parameters (temperature and particle size) towards product yield by applying slow mode pyrolysis.

iv) To identify the chemical component presents in the liquid-oil produced with different pyrolysis condition.

1.4 Scopes of research

The scopes of this research are:

i) The biomass samples undergo characterization analysis such as proximate, ultimate, and component analysis to check the properties of *Imperata cylindrica* by using Thermogravimetric analysis (TGA), CHNS/O analyzer, bomb calorimeter and several analytical procedures.

ii) Running pyrolysis process in a batch reactor using slow mode pyrolysis with constant heating rate of 22 ºC . min^{-1} by varying two process parameter (process temperature between 450 ºC to 600 ºC and particles size of 0.25 to 1 mm).

iii) Analyzing the chemical compound in the liquid oil obtained from the pyrolysis process by using GC-MS and the water content using Karl Fischer titration for each sample.

1.5 Hypothesis

The hypothesis for this research includes;

i) Slow pyrolysis of *Imperata cylindrica* were expected to produce 40 %, 30 % and 30 % of solid, gas and liquid yield, respectively.

ii) Employing larger particle size were expected to produce more amounts of liquid and solid yield and resulting less amount of gas yield compared to lower particle size. The increases of temperature in pyrolysis are expected to produce more gases while solid and liquid yield will be decreased.

iii) From the chemical characterization using mass spectrometry (GC–MS), the pyrolysis liquid products may contains substantial proportion of water, some solid particles and some organic compounds that belong to acids, alcohols, ketones, aldehydes, phenols, ethers, esters, sugars, furans, nitrogen derivatives and others possible compounds.

1.6 Significant of Research

The significant of this research is mainly to study the potential of *Imperata cylindrica* or cogon grass as a biomass feedstock for slow pyrolysis process. This biomass was chosen due to its abundant resources which can be found everywhere and easy to grow.

Today, research focuses more on fast pyrolysis process in producing more bio-oil but in this study, slow pyrolysis were run in a batch fixed bed reactor to analyses the product distribution yield by varying the biomass particle size and pyrolysis temperature. This would contribute to the new information and knowledge for this scope of research.

1.7 Thesis Organisation

Chapter 1 contains the introduction of the research and describe the objectives and the significance of this study. In Chapter 2, literature review and vigorous study about the biomass (*Imperata cylindrica*), type of pyrolysis, the characterization of biomass and properties of bio-oil were highlighted in this chapter. Research methodology in Chapter 3 were describe about the procedure of the characterization of the biomass, the instrument used, the rig set-up, the experimental procedure for the pyrolysis, and also the analysis of the product (bio-oil) using GC-MS. Chapter 4 presents the result and discussion of the characterization of the biomass in comparison with the previous study (slow pyrolysis of rice husk) together with the product distribution yield for slow pyrolysis process in a fixed bed reactor. The result of water content and component identification using GC-MS were also been discussed in this chapter. In the last Chapter which is Chapter 5, conclusion and recommendation were discussed to summarize and improving this research in future.

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