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MICROWAVE-ASSISTED PYROLYSIS FOR CONVERSION OF KARANJA SEED TO BIO-OIL

Charmaine Joanna anak Stephen Cornet

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MICROWAVE-ASSISTED PYROLYSIS FOR CONVERSION OF KARANJA SEED TO BIO-OIL

CHARMAINE JOANNA ANAK STEPHEN CORNET

A dissertation submitted in partial fulfilment
of the requirement for the degree of
Bachelor of Engineering with Honours
(Mechanical and Manufacturing Engineering)

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To my biggest strength and love, beloved family and friends.

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ABSTRACT

The growing needs of fuels has been a major problem not only towards the society but as well to the environment. In order to satisfy this energy demand and needs without damaging the Mother Nature and affect the whole ecosystem, bio oil from biomass has been introduced as a potential biofuel that can be used as a replacement for conventional fuel. Microwave-assisted pyrolysis has been recognized as one of the most effective and feasible thermochemical process that able to convert biomass feedstock into three main products which are bio char, bio oil and bio gas. The bio oil obtained from the pyrolysis process are used as an alternative fuels and also other chemical products. In this study, conversion of non-edible seeds which is Pongamia Pinnata, also known as Karanja seed into bio oil had been performed by using two method which are microwave pyrolysis and also conventional pyrolysis. A modified household microwave oven of 800W and microwave pyrolysis reactor was used for the microwave pyrolysis method whereas for conventional pyrolysis, a modified heating mantle apparatus was used to pyrolyzed Karanja into bio oil. Different pyrolysis temperature and time was applied to both techniques to achieved the conversion of bio oil. Finding shows that the yield of bio oil for microwave heating at 350°C and 400°C ranged from 10.70 wt% - 25.60 wt% within 6, 9 and 12 mins. Meanwhile, the yielding for conventional heating with the same temperature as microwave but different pyrolysis time (30, 40 and 50 mins) are recorded from 15.42 wt% - 36.07 wt%. The FTIR results indicated that there are a vast range of functional groups such as carboxylic acids, ketones, alcohols and aldehydes are present in the bio-oils. Moreover, the highest calorific value recorded for both microwave and conventional pyrolysis are 21.2341 MJ/kg and 20.9849 MJ/kg respectively. The pH value also shows that the bio-oils are in the acidic properties as it is the range of 5 to 6.

ABSTRAK

Keperluan bahan api yang semakin meningkat telah menjadi masalah utama bukan sahaja kepada masyarakat tetapi juga kepada alam sekitar. Untuk memenuhi permintaan dan keperluan tenaga tanpa menjejaskan keseimbangan ekosistem, minyak bio yang diperbuat daripada biomas telah diperkenalkan sebagai *biofuel* kerana berpotensi mengganti bahan api konvensional. Pirolisis gelombang mikro merupakan salah satu proses termokimia yang paling efektif untuk menghasilkan char bio, minyak bio dan gas bio daripada bahan mentah biomas. Minyak bio yang diperoleh daripada proses pirolisis bukan sahaja boleh digunakan sebagai bahan bakar alternatif malah ia juga boleh digunakan sebagai produk kimia lain. Dalam kajian ini, minyak bio telah dihasilkan daripada *Pongamia Pinnata*, atau juga dikenali sebagai biji Karanja melalui dua kaedah yang berlainan iaitu proses pirolisis gelombang mikro dan proses pirolisis konvensional. Proses pirolisis gelombang mikro telah dilakukan dengan menggunakan sebuah ketuhar gelombang mikro 800W manakala proses pirolisis konvensional telah dilakukan dengan menggunakan radas mantel pemanasan yang telah diubahsuai. Untuk kedua-dua proses pirolisis ini, pembolehubah yang telah dimanupulasikan adalah suhu pirolisis dan masa pirolisis. Hasil daripada kajian ini menunjukkan bahawa 10.70% berat hingga 25.60% berat minyak bio telah berjaya dihasilkan menggunakan kaedah pemanasan gelombang mikro pada suhu 350°C dan 400°C dalam tempoh 6, 9 dan 12 minit. Dengan menggunakan suhu yang sama, kaedah pemanasan konvensional pula telah menghasilkan 15.42% berat - 36.07% berat minyak bio dalam tempoh 30, 40 dan 50 minit. Analisis FTIR telah menunjukkan bahawa terdapat banyak kelompok berfungsi seperti asid karboksilat, keton, alkohol dan aldehid dalam minyak bio yang telah dihasilkan tersebut. Selain itu, nilai kalori tertinggi yang dicatatkan untuk gelombang mikro dan pirolisis konvensional adalah 21.2341 MJ/kg dan 20.9849 MJ/kg. Nilai pH 5 hingga 6 juga menunjukkan bahawa minyak bio yang telah dihasilkan tersebut mempunyai sifat asid.

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LIST OF ABBREVIATIONS

HTL	-	Hydrothermal liquefaction
CO_2	-	Carbon dioxide
FTIR	-	Fourier transform infrared spectroscopy
GHz	-	Giga hertz
MSW	-	Municipal solid waste
W	-	Watt
HHV	-	Higher heating value
LHV	-	Lower heating value
C	-	Carbon
H	-	Hydrogen
N	-	Nitrogen
S	-	Sulphur
CO	-	Carbon monoxide

CHAPTER 1

INTRODUCTION

1.1 Background study

Energy is the most vital and important driving force in this modern, fast track world. It is part of the necessity in human's life which compromises variety of sectors such as economic, transportation, industry, agriculture and also as part of power generation (Ingole et al., 2016). With the usage of energy, it helps to enhance the living standard of society in terms of social and economic (Ali et al., 2017). Energy can be further divided into two constituent sources which are renewable and non-renewable energy. Biomass is part of the renewable energy along with solar, hydroelectric, wave, geothermal and wind energy.

Biomass is a form of energy that comes from living species such as plants and animals. It is also formed from waste materials, food, and also wood. Biomass is viewed as renewable energy source as it can reproduce and replenish to ensure its sustainability. Not only that, the resources are often available and infinity. This is one of its significant attractions as a wellspring of vitality. In contrast to renewable energy, although fossil fuels such as coal and gas are convenient sources of energy; however, it takes longer time to be created, and the sources will definitely run out in the near future due to high energy demand and consumption. Therefore, alternative energy resources such as renewable energy are being further develop and research to cater to society needs and demands especially in a long run.

The first on demand energy that humans starts to utilize is biomass and its position as a primary supply of energy rely on the topographical and economics conditions (Basu, 2010). The two main forms of energy derived from biomass is heat and electricity. This bioenergy that are produced from biomass can be a substitute of fossils fuels especially in the chemical industry to produced heat, electricity and liquid fuels for transportation sector (Lorenz & Lal, 2018). Biomass is the only renewable energy source that can supply bioenergy in the form of solid, liquid and gaseous fuels. There are two major ways that can help in achieving the process of conversion from solid biomass to liquid and gaseous fuels which are biochemical and thermochemical processes (Yildiz et al., 2011).

The most convenient way to develop and produce liquid bio fuels is through the process of thermochemical process. Thermochemical process involves two main paths which are pyrolysis and hydrothermal liquefaction. Pyrolysis is a process that involves the conversion of biomass feedstock to produce three main products which are in the state of solid, liquid and gas by using high temperature with the absence of oxygen. Meanwhile, HTL is a direct liquefaction that is conducted under gradually increasing pressure and temperature to ensure that water is in either two state which is liquid or supercritical. It can also be conducted with or without the presence of catalyst. It is a process that converts high moisture biomass into crude bio oil and other chemicals that can be used for combustion or transportation (Xiu & Shahbazi, 2012). Whereas for pyrolysis process, pre-treatment such as drying is required as moisture content in the feedstock could affect the heating rate.

Bio-oil is one of the end products from pyrolysis process apart from the production of bio char and bio gas. It is known as pyrolysis oil that is dark brown in colour which have a distinctive and pungent smoky fragrance and are free flowing. There are several properties of bio oils which resulted from the chemical composition of the oils which differs it from the conventional petroleum-derived oils. Some of the physical properties includes oxygen content, water content, viscosity, corrosiveness and lastly combustion behavior. These physical properties of bio oils are different and it depends on the feedstock characteristics and compositions. Compared to the traditional biomass fuels, bio oil has a higher efficiency energy production especially for the generation of heat and power as well as for fuel transportation. It is also viewed as carbon dioxide (CO_2) neutral due to lower sulfur and nitrogen gas content which can lead to lower greenhouse gas emissions (Czernik & Bridgwater, 2004). Countries that produce great number of organic wastes has the potential to produce and develop this renewable energy source locally at their countries. Thus, creating more environmentally friendly bio-oil that are safer and cleaner which causes less pollution in the world. Bio-oil can serve as an alternative fuel for various applications such as boilers, furnaces, engines and turbines. Mostly for power, heat and electricity generation. However, it is still cannot be used as a transportation fuels due to undesirable properties and needed further upgrading to be used as an alternative for petroleum fuel (Xiu & Shahbazi, 2012).

1.2 Problem statement

Rapid growth and urbanization in the world have led to an increasing of the world population, so as the energy demand and needs. With the rise of energy consumption, there has been a growing concern regarding the usage of fossil fuels in the world. The consumption of fossil fuels is gradually increasing along with the development of industrial and quality of life. The continuous consumption of fossil fuels has leads to direct impact towards the environment which causes global climate change and threat towards human health. Therefore, society are trying to seek and exploit for more alternative energy sources that could combat the oil crisis as the demands for cleaner and safer fuel are escalating and the authority need to keep and preserve these natural resources before it faces the threat of extinction.

Besides that, increased usage of biomass also has a potential negative impact on the environment as well as the people. It could possibly decrease the food security, increase water pollution and threaten conservation areas (Field, Campbell & Lobell, 2007). Competition among the farmers for land and feedstock will be greater once they notice that the commercialize of bio oils are getting higher and it is a good source of income. This will lead to shortage of food supplies as most will change from food crop to bio oil crop instead. It will also affect the economy due to increase price of food globally. Deforestation will also increase due to greater demand for bio oil crop (Field, Campbell & Lobell, 2007).

Other than that, environmental impact due to global warming, thinning of ozone layer and rising of sea levels are also at an alarming level. Emission of carbon dioxide, sulfur and nitrogen due to the usage and burning of conventional fuel especially in an internal combustion engine can have great influence towards climate change, animals and the ecosystem. Formation of acid rain, increase in carbon footprint and greenhouse gases are part of the environmental effect. Hence, by converting biomass into bio fuels, it can generate less nitrogen, sulfur and carbon emission to the atmosphere as biomass plants contains insignificant amount of sulfur and it absorbs the carbon from the surrounding by photosynthesis.

One of the routes for converting biomass into bioenergy is through the pyrolysis process. Microwave heating technique provide instantaneous heat and offers rapid heating to convert agricultural waste into a value-added product. Salema and Ani (2010) reported that conventional heating methods has a lot of drawbacks compared to microwave pyrolysis. The limitations that conventional heating can be referred such as heat losses to surrounding, heat transfer resistance and also damages to the reactor wall due to electric heating. Long heating duration also required in conventional heating as the heat distribution was slower and not uniform which resulted in an undesired or secondary reaction towards the biomass feedstock. Hence, microwave-assisted pyrolysis is much preferred and is being developed to be use as an alternative for the pyrolysis of biomass.

1.3 Objectives of Research

The objective of this research is to produce bio oil from the Karanja seed by using the method of pyrolysis. To achieve these, a study was carried out with the following objectives:

i. To extract bio-oils from Karanja seed by using the method of microwave-assisted pyrolysis and conventional heating pyrolysis.
ii. To characterize the bio-oil production from microwave and conventional pyrolysis.
iii. To fabricate and develop equipment for the process of microwave-assisted and conventional heating pyrolysis.

1.4 Significance of Research

This research is conducted to study the properties and quality of the bio-oil produced by using Karanja seed as the biomass feedstock. Bio-oil can be used as an alternative for the production of bio fuels which can be used in industrial sector as well as power and heat generation. The chosen method for this research is by using the process of fast pyrolysis which will be assisted by using a microwave. There will be three techniques used to evaluate and analyse the chemical composition that are present in the bio-oil which are Fourier Transform Infrared Spectroscopy (FTIR), bomb calorimeter and pH testing.

CHAPTER 2

LITERATURE REVIEW

2.1 Karanja

2.1.1 History of Karanja

Karanja tree is a species of tree that existed in the pea family. It is significantly known as Pongamia pinnata and it comes from the family of Leguminasae where it originated from India and widely grow throughout the Indian subcontinent and Southeast Asia including China, Japan, Malaysia and Australia (Saksule & Kude, 2012). It is a medium sized evergreen tree that produce oil bearing seed, however the seeds are non-edible like jatropha and mahua. Not only it produced seeds that have useful oil but it also shows tolerance towards high temperature especially during droughts. Planted as an ornament due to great canopy shade that are equally spread and with an average height of 15-20 meters, Karanja can be grown in humid and subtropical environments which resulted in huge availability of this particular species in most tropical climate countries (Mamilla, Mallikarjun & Rao, 2011).

Furthermore, this tree has a high tolerance towards salinity hence it can grow in various soil conditions and types ranging from salty, alkaline, sandy, stony and waterlogged soils (Halder et al., 2014). It is said that this medium sized tree can mature within 4 to 7 years and the seed obtained from one tree can yield about 9 to 90 kg of seed depending on the tree size. Karanja pods are elliptical shaped that contains one or two kernels that are the sized of pea which has the same shape as a kidney and are in reddish brown colour. These Karanja seeds bear 30% - 40% of oil in it and the trees are one of the few nitrogen-fixing trees (Saksule & Kude, 2012). As mentioned by Bobade and Khyade (2012), Karanja oil can reach 13500 million tonnes of productions per annum however only 6% of the oil is being utilized by society. Besides that, this Karanja oil contain toxic flavonoids and furanoflavones which makes it non-edible hence it can be applied for the production of biodiesel (Mamilla, Mallikarjun & Rao, 2008).

In the olden days especially India and other neighboring division, Karanja plant has been used as a traditional medicine due to its properties that are anti-inflammatory, antifungal and antibacterial. It acts as a medicinal plant that helps to treat various kinds of ailments such as abscess, skin disease, tumors, diarrhea and also it is used as an antiseptic and blood purifier (Kumar, Kumar & Prakash, 2012). However, with the advance technology and growing society, Karanja plant has been seen as a possible and alternative source of bio fuel especially for the production of biodiesel (Atabani et al., 2012)

Figure 2.1 Karanja tree with the fruits (Saksule & Kude, 2012)

2.1.2 Uses of Karanja

Karanja plant are not only suited for the production of oil but it also has great potential on various of applications. It is not only restricted to the seeds but other parts of the trees such as the wood, oil, leaves, kernel, de-oiled cake, fruit hull and also the root and bark.

Wood from a Karanja tree is used as fuel especially in remote area in Bangladesh however, the timber is said to be in poor quality due to its low durability and high tendency to break during sowing (Saksule & Kude, 2012). The timber is also at high risk for insect attack therefore lessen the properties of the timber itself. Hence, it is only used for cabinet making, cartwheels, tools handle and many more. Other than that, the ashes produced from burning the timber is commonly used for dyeing (Halder, Paul & Beg, 2014).

Oil that is produced during the seed extraction is considered as non-edible and it produces undesirable odour as well as pungent taste hence it is only used for commercial purposes. The oil is commercially promoted as medicine, cooking fuel, lubricant, biodiesel and also for soap making (Bobade & Khyade, 2012).

Furthermore, parts of the Karanja tress such as flowers, fruits and seeds can be used to treat numerous sickness. As for instance, the flowers were used as hemorrhoids treatment whereas the fruits can help in treating ulcers and abdominal tumors. Other than that, the seed powder is used to relieves fever, bronchitis and cough. Besides that, other parts of the tree consist of leaves, bark and root is being utilized as well to be in aid for inflammation relieves, treating cold and coughs, wounds and also as a remedy for digestive systems (Bobade & Khyade, 2012). This specific plant proved to be useful in many sectors by utilizing every parts of its tree for mankind to explore and exploit more of it.

2.2 Pyrolysis

Thermal conversion of waste biomass into beneficial energy sources to replace non-renewable energy with high quality products involves a wide range of processes. Few of the thermal decomposition processes are gasification, combustion and pyrolysis (Nomanbhay et al., 2017). Pyrolysis is one of the recognized thermochemical conversion of biomass into bio oil, bio gas and bio char. The word pyrolysis comes from a Greek word which brings the meaning of “pyro” as fire while “lysis” means degradation. Pyrolysis converts biomass into useful products such as biofuel and the pyrolysis process involve organic compounds degradation by using high temperature with the absence of oxygen which resulted in the production of liquid, solid and gaseous substances which can be used as an energy source (Xiu & Shahbazi, 2012).

Neves et al (2011) mentioned that pyrolysis can be categories into three reactions which are drying, primary pyrolysis and secondary pyrolysis. In the drying stage, feedstock will undergo evaporation to remove the moisture content in the sample before it can proceed to the next step which is primary pyrolysis stage. For the next stage, the feedstock undergoes primary pyrolysis where a non-volatile solid called bio char will be produced through the decomposition of biomass at which the feedstock experiences thermal scission as a result of molecular deterioration on the chemical bonding. Further increased on the temperature during primary pyrolysis will allow the feedstock to experience secondary pyrolysis. In this stage, the volatile matter that are released will undergo condensation where it will turn into pyrolysis oil or commonly known as bio oil and also non-condensable gas which is the bio gas (Nomanbhay et al., 2017).

There are three main operating parameters that affect the characteristics of the process such as heating rate, pyrolysis temperature and residence time. Each parameter played a major role in determining the end product of the pyrolysis process (Zhang et al., 2017). Pyrolysis will always produce three products mainly biochar, bio-oil and bio gas however the ratios can vary by adjusting the parameters stated earlier. Higher pyrolysis temperature and short residence time favors the conversion of biomass to bio gas whereas lower temperature and longer residence time increase the production of biochar. Moreover, the most suitable and optimum parameters to produce bio oil is moderate pyrolysis temperature with short residence time (Ingole et al., 2016). Pyrolysis can be divided into three parts depending on its operating conditions which are slow pyrolysis, fast pyrolysis and flash pyrolysis.

2.2.1 Slow Pyrolysis

Slow pyrolysis or commonly known as conventional pyrolysis is a process that applies low temperature and heating rate with long residence time. It is the traditional way to produce charcoal with the main objective to yield fuel that does not produce any smoke (Kundu et al., 2018). This process allows the feedstock to undergo slow devolatilization where biochar and tar will be produced. Biochar are now developed and tested to be used as a soil amendment to enhance the characteristics of the soil and to reduce the emission of greenhouse gases where it acts as a carbon sink (Muray, Resende & Luo, 2014). Zhang et al (2017) stated that for slow pyrolysis process that favors the production of bio char has a longer residence time of more than 450 seconds and relatively low temperature range of about 300°C till 700°C. Not only that it is characterized by residence time and temperature but also pyrolysis heating rate. Heating rate for slow pyrolysis is less that $1℃/s$.

2.2.2 Fast Pyrolysis

Fast pyrolysis uses very high temperature on the feedstock without the presence of oxygen to let it vaporized at which the vapour condensed into liquid mainly called pyrolysis oil or bio-oil. Pre-processing of the feedstock before the pyrolysis process are necessary to avoid any major interference in the stability, calorific value, pH, and also corrosiveness of the product obtained. Some of the essential pre-processing steps include drying, reduction of particle size, grinding and moisture content analysis (Kundu et al., 2018). Particle size has a major impact on the heating rate of the feedstock because it enables the pyrolysis process to be more uniform and rapid. Neves et al (2011) stated that when larger particles size are used for the pyrolysis, the liquid yield produced lower result as it have a direct influence on the volatiles during secondary reactions. Fast pyrolysis most likely favors the production of bio oil due to its short residence time of 0.5 until 20 seconds with moderate temperature range of 550°C -1250°C and high heating rate for about 10-300°C/s (Zhang et al., 2017).

Fast pyrolysis is more desirable as it produces mostly bio oils together with minimal amount of bio char and bio gas which has a potential to be marketed at a higher value. Not only that, this technology has a higher energy efficiency compared to slow and flash pyrolysis as well as relatively low cost of investments (Guedes, Luna & Torres, 2018). The end product produced from fast pyrolysis also has a very wide range that can be used in many applications namely heat and power generation, liquid fuels and also for raw chemical products. Although it can be applied to many applications, bio oil is still not suitable as transportation fuel due to few undesired properties such as high viscosity, low heating value, corrosiveness, and thermally unstable (Murray, Resende & Luo, 2014)

2.2.3 Flash Pyrolysis

Flash pyrolysis is one of the process that can achieve relatively high bio oil yield which can go up to 75% (Jahirul et al., 2012). The process is similar with fast pyrolysis but the difference is the amount of oil yield. In comparison with fast pyrolysis, flash produced more bio oil yield than fast pyrolysis. However, there are some limitation towards this particular process where it has poor thermal stability, high corrosiveness in the oil, chemical instability of the bio oil and presence of substances in the oil (Kundu et al., 2018).

Flash pyrolysis has the highest heating rate, shortest residence time and high temperature range which is above 1000°C/s, below 0.5 second and about 800 - 1300°C respectively (Zhang et al., 2017). The particle size should be small when uses flash pyrolysis due to the rapid heating range.

Table 2.1 Operating parameters and products yield for pyrolysis process (Jahirul et al., 2012)

Pyrolysis Process	Solid Residence Time (s)	Heating Rate (K/s)	Particle Size (mm)	Temp. (K)	Product Yield (%)		
					Oil	Char	Gas
Slow	450–550	0.1–1	5–50	550–950	30	35	35
Fast	0.5–10	10–200	<1	850–1250	50	20	30
Flash	<0.5	>1000	<0.2	1050–1300	75	12	13