HORSE MANURE DERIVED BIOFUEL PRODUCTION VIA MICROWAVE INDUCED PYROLYSIS

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ABSTRACT

Uncontrollable amount and unsystematic management of generated wastes due to the ever-increasing global population of human and animal have led to the alarming environmental pollution and health issues. Thus, waste-to-product transformation process is gaining popularity in recent years as this allows for the conversion of zerovalue waste into beneficial end products. Horse manure (HM) is deemed to be a feasible feedstock for waste-to-energy transformation through thermochemical conversion. Conventional HM management methods, such as natural composting, have led to undesirable environmental pollution. The aim of this study was to determine the feasibility of HM to be converted into bio-products through microwave pyrolysis. The decomposition and kinetics behaviour of HM were investigated through thermogravimetric analyser (TGA) at temperatures of 27-900 °C and heating rates of 1-10 °Cmin⁻¹. The feedstock experienced rapid decomposition at temperatures between 190 °C and 400 °C under oxygen-free environment. Such a decomposition process was found to be endothermic and endergonic in nature, giving an activation energy of approximately 148.5-300.1 kJ/mol. The finding suggested that pyrolysis is the preferred thermal conversion pathway. A lab-scale microwave-heated pyrolysis reactor was then setup for the decomposition process of HM whilst collecting end products in the form of solid (bio-char), liquid (bio-oil) and gas (bio-gas). The produced bio-gas consists up to 70.2 vol% of syngas (H₂ and CO) along with CH₄ (12.6 to 23.4 vol%), making it a gaseous fuel candidate for heat/power generation. Although the bio-oil is made up of oxygenated compounds that resulted in low heating value (6.2-15.8 MJ/kg), its high phenolic content (up to 79 wt.%) remains useful for application as bio-chemical product. The bio-char derived from the microwave pyrolysis of HM is highly porous (surface area of up to 698.4 m^2g^{-1}), having potential as bio-adsorbent products. The bio-char produced also gained in heating value (up to 111.1%), indicating solid fuel-like properties. The optimum pyrolysis process parameter in yielding desired amount of end products was measured statistically through a full factorial design (FFD). The results indicated AC/HM ratio as the most influential factor, followed by temperature and carrier gas flow rate. Optimization of the end products suggested that a process condition with AC/HM ratio of 2.0, N₂ flow rate of 0.5 Lmin⁻¹ and temperature of 550 °C, giving end product yields of 13.5 wt.% of bio-char, 32.5 wt.% of bio-oil and 39.2 wt.% of bio-gas. Lastly, a life-cycle-analysis was also conducted and the results show that HM feedstock is more environmentally friendly as compared to swine manure, when both are processed through pyrolysis. The management of HM through pyrolysis is also found to be the preferred pathway as compared to incineration, anaerobic digestion and natural composting. Overall, the study demonstrated that microwave-induced pyrolysis has the potential to thermochemically-convert HM into beneficial end products. Coupling this with the positive outcome from the life-cycle-analysis, it could be further summarised that HM is indeed a viable feedstock to be considered for valorisation purposes through microwave-induced pyrolysis.

ABSTRAK

Populasi manusia dan haiwan yang semakin meningkat telah mengakibatkan penambahan jumlah sisa buangan. Pengurusan sisa buangan yang tidak sistematik telah menyebabkan masalah pencemaran alam sekitar dan akan mempengaruhi kesihatan manusia. Oleh itu, proses transformasi sisa ke bio-produk semakin popular di kalangan ahli sains kerana ini memungkinkan transformasi sisa yang bernilai sifar kepada produk yang bermanfaat. Najis kuda (HM) adalah sejenis sisa yang mempunyai potensi untuk diubah menjadi sumber tenaga melalui penukaran therma-kimia. Secara konvensional, pengurusan HM dijalankan melalui kompos semula jadi dan kaedah ini telah mengakibatkan pencemaran alam sekitar. Tujuan kajian ini adalah untuk menentukan penukaran HM kepada bio-produk melalui proses pirolisis gelombang mikro. Tingkah laku penguraian dan kinetik HM diselidik melalui penganalisis termogravimetrik (TGA) pada suhu 27-900 °C dan kadar pemanasan 1-10 °Cmin⁻¹. Kadar penguraian yang tertinggi adalah pada suhu antara 190 °C sehingga 400 °C di bawah persekitaran bebas oksigen. Proses penguraian sedemikian bersifat endotermik dan endergonik. Di samping itu, tenaga pengaktifan yang diperlukan adalah sebanyak 148.5-300.1 kJ/mol. Data menunjukkan pirolisis merupakan kaedah yang sesuai untuk memproses HM. Sebuah reaktor pirolisis gelombang mikro berskala makmal telah dihasilkan untuk proses penguraian HM. Produk akhir dalam bentuk pepejal (bioarang), cecair (bio-minyak) dan gas (bio-gas) telah dikumpulkan. Bio-gas yang dihasilkan terdiri daripada 70.2 vol% syn-gas (H2 dan CO) dan CH4 (12.6 hingga 23.4 vol%). Ia mempunyai potensi sebagai bahan bakar untuk penjanaan haba / tenaga. Walaupun bio-minyak mempunyai sebatian beroksigen yang tinggi menghasilkan nilai pemanasan yang rendah (6.2-15.8 MJ/kg), namun kandungan fenoliknya yang tinggi (hingga 79 wt.%) tetap berpotensi untuk digunakan sebagai produk bio-kimia. Bioarang mempunyai luas permukaan yang tinggi (698.4 m²g⁻¹) dan berpotensi dijadikan produk bio-penyerap. Bio-arang yang dihasilkan juga mengalami kenaikan dalam nilai pemanasan (hingga 111.1%), menunjukkan potensi sebagai bahan bakar dalam bentuk pepejal. Parameter optimum untuk menghasilkan jumlah produk yang tertinggi diukur secara statistik melalui reka bentuk faktorial penuh (FFD). Hasil kajian menunjukkan nisbah AC/HM sebagai faktor yang paling berpengaruh, diikuti oleh suhu dan kadar aliran gas. Pengoptimuman produk akhir mencadangkan bahawa proses pirolisis perlu dijalankan dengan nisbah AC/HM 2.0, kadar aliran N₂ 0.5 Lmin⁻¹ dan suhu 550 °C. Hasil produk akhir dianggarkan sebanyak pada 13.5 wt.% bio-arang, 32.5 wt.% biominyak dan 39.2 wt.% bio-gas. Analisis kitaran hidup juga dijalankan dan diputuskan bahawa ia lebih mesra alam berbanding dengan pirolisis sisa khinzir. Pengurusan HM melalui pirolisis dilaporkan sebagai cara yang lebih mesra alam berbanding dengan pembakaran, pencernaan anaerobik dan kompos semula jadi. Secara keseluruhannya, kajian menunjukkan bahawa pirolisis yang dijalankan melalui gelombang mikro berpotensi untuk mengubah HM kepada produk akhir yang lebih bermanfaat. Tambahan dapatan kajian dari analisis kitaran hidup, ia dapat disimpulkan bahawa HM sememangnya sejenis bahan sisa yang dapat dinaik taraf kepada produk yang berguna melalui proses pirolisis dengan gelombang mikro.

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## LIST OF ABBREVIATIONS

AC	-	Activated Carbon
AKTS	-	Advanced Kinetics and Technology Solution
ANOVA	-	Analysis of variance
AP	-	Acidification potential
BBD	-	Box-Behnken design
BET	-	Brunauer-Emmett-Teller
CCD	-	Central composite design
DAEM	-	Distributed Activation Energy Model
DF	-	Degree of freedom
DOE	-	Design of experiments
DTG	-	Derivative Thermogravimetry
EDX	-	Energy dispersive X-ray
EIA	-	Energy Information Administration
EP	-	Eutrophication potential
FESEM	-	Field emission scanning electron microscopy
FFD	-	Full Factorial design
FWO	-	Flynn-Wall-Ozawa
G	-	Graphite
GC-MS	-	Gas Chromatography Mass Spectroscopy
GC-TCD	-	Gas Chromatography Thermal Conductivity Detector
GHG	-	Greenhouse gas
GWP	-	Global warming potential
HDPE	-	High-density polyethylene
HHV	-	Higher heating value
HM	-	Horse Manure
HTL	-	Hydrothermal liquefaction
HTP	-	Human toxicity potential
KAS	-	Kissinger-Akahira-Sunose
LCA	-	Life cycle analysis
LHV	-	Lower heating value

MIP	-	Microwave-induced pyrolysis
MSW	-	Municipal solid waste
NREL	-	National Renewable Energy Laboratory
ODP	-	Ozone depletion potential
PAHs	-	Polycyclic aromatic hydrocarbons
PID	-	Proportional-Integral-Derivative
POCP	-	Photo-oxidant formation potential
R		Universal gas constant
RC	-	Residue char
RSM	-	Response surface methodology
SB model	-	Šesták and Berggren model
SMS	-	Spent mushroom substrate
TG	-	Thermogravimetry
TGA	-	Thermogravimetric analysis
TNB	-	Tenaga National Berhad
U.S.	-	United States

## LIST OF SYMBOLS

α	-	Rate of mass loss
β	-	Heating rate
ε"	-	Dielectric loss factor
ε'	-	Dielectric constant
f(a)	-	Kinetics reaction model
da/dt	-	Conversion rate
$\tan \delta$	-	Loss tangent
λ ₀	-	microwave wavelength in free space
$Sj^2$	-	Residual sum of square
$m_0$	-	Initial mass
$m_{\rm f}$	-	Final mass
$E_{\alpha}$	-	Activation energy
$t_{\alpha}$	-	Time to reach specific conversion
$\mathbf{F}_{j}$	-	F-test statistical analysis
$D_p$	-	Penetration depth
K _B	-	Boltzman constant
А	-	Pre-exponential factor
с	-	velocity of light in free space
f	-	frequency of the electromagnetic wave
h	-	Plank constant
k	-	Constant of decomposition rate
Т	-	temperature
t	-	time

#### **CHAPTER 1**

#### **INTRODUCTION**

#### 1.1 Introduction

Modernisation and technological advancements have brought the world into a new era of information and technology. However, the comfort of living brought by such technological advancements has led our planet to be drained of its natural resources, particularly for the use of energy generation. The U.S. Energy Information Administration reported that the world energy consumption has increased by more than 50% from the 1990s to the present time as shown in Figure 1.1. It has been predicted that this trend will continue into the next decade (EIA, 2017).

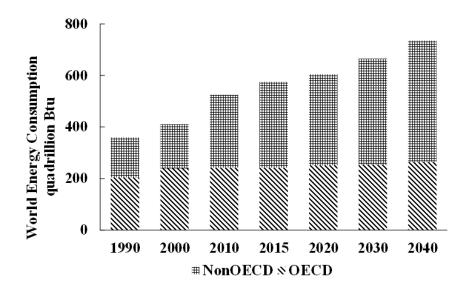


Figure 1.1 World Energy Consumption. Adapted from (EIA, 2017)

Petroleum has thus far been the main fuel supply for energy generation. Based on the current state of crude oil production, the energy demand could no longer be met by relying solely on this fuel source (Andruleit et al., 2015). Therefore, alternative fuel resources for energy generation are being explored. Figure 1.2 indicates that renewable energy will experience a slow but steady growth due to its cleaner power generation and never ending supplies (EIA, 2017). Renewable energies can also be sourced from the sun (solar energy), wind (wind energy), water (hydro energy), earths' heat (geothermal energy) and biomass.

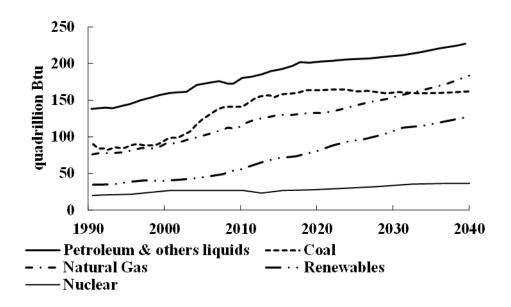


Figure 1.2 World Energy Consumption by Energy Source. Adapted from (EIA, 2017)

Among the above-mentioned renewables, solar energy will not be available all the time and can only be harvested at particular periods during the day. Moreover, weather changes will pose as the main obstacle in solar energy harvesting. On the other hand, wind energy can only be harvested at certain parts of the world, mostly along high hills area or seaside, where wind speed is sufficiently strong. Even though hydro energy can be harvested all-year-round, building of a dam has been known to lead to undesired deforestation and significant changes in landscape. Another alternative is to resort to geothermal energy, which is only available in limited regions on earth (K ád ár, 2014; Mohtasham, 2015).

Of all the above mentioned renewable energies, biomass remains as the only source, which is available to all parts of the world in abundance and varieties. Biomass comes in various forms, comprising of woods, crops, garbage, manure, alcohol fuels and landfill gas. Energy extraction from biomass waste can be done through various conversion methods. Among them are thermochemical conversion and biochemical conversion techniques.

Thermochemical conversion methods include: 1) combustion, 2) pyrolysis, 3) torrefaction and 4) gasification. These methods involve the usage of heat, with or without the presence of oxygen, to convert biomass into valuable end-products. On the other hand, biochemical conversion methods include: 1) anaerobic/aerobic digestion and 2) fermentation. These methods mainly involve the usage of enzymes, bacteria or microorganism to breakdown biomass feedstock in producing valuable end-products. Both methods generate end-products in the form of solid, liquid and/or gas. Among these conversion methods, pyrolysis has been demonstrated to be effective in producing value-added end-products in the form of solid (bio-char), liquid (bio-oil) and/or gas (bio-gas/syngas) (Prithiraj & Kauchali, 2017). Besides this, pyrolysis has also been shown to emit smaller quantity of pollutants, such as  $SO_x$ ,  $NO_x$  and  $CO_x$ , when compared with combustion and gasification thermochemical conversion methods (Cao et al., 2014).

Pyrolysis can also be referred to as the thermal decomposition of material under intense heat within an inert atmosphere. There are numerous chemical reactions that take place during pyrolysis, which complicates the prediction of the thermochemical conversion behaviour and the reactivity of different feedstock. To have a more detailed insight into the thermochemical conversion process during pyrolysis reaction, a kinetics analysis is typically conducted on the sample feedstock (Balasundram et al., 2017). The data obtained through such a kinetics analysis is capable of providing useful technical insights for designing and building an effective pyrolysis rig. For this purpose, the thermo-gravimetric analysis (TGA) can be used to determine kinetics parameters (Cai et al., 2018). Through this analysis, activation energy, which refers to the minimum energy to initiate chemical reaction of the sample, could be determined.

The present study aims to first explore the valorisation of horse manure (HM) through pyrolysis using thermogravimetric analysis in assessing its potential as a beneficial feedstock in bioenergy harvesting. Thermal decomposition kinetics of HM is then determined through iso-conversional method. A microwave pyrolysis rig is then

designed for the thermal decomposition of HM under varying pyrolysis process parameters (e.g. temperature, carrier gas flow rate and catalyst ratio). A statistical analysis is then carried out to ascertain a mathematical model that correlates thermal decomposition product yield and pyrolysis process parameters, such as operating temperature, carrier gas flow rate and catalyst ratio. Finally, the end-products will be characterised accordingly. Based on the proposed quantitative and qualitative analysis, relevant and suitable applications for the thermally decomposed HM end-products would then be identified.

#### **1.2 Problem Background**

Biomass and bio-wastes have been studied as renewable fuel sources due to its carbon neutral properties (Lam et al., 2018; Ng et al., 2017; Samiran et al., 2016). Animal manure is an abundant biomass waste source, where its amount can be totalled up to 2.62 x  $10^{13}$  kg/year on a worldly basis. These values far exceed the amount produced by human population (Penakalapati et al., 2017). Existing manure managing facilities have been deemed to not be capable of coping with this overwhelming amount of manure. A report by The Food and Agriculture Organization of the United Nations stated states that about 75% of manure produce globally were left on permanent meadows and pastures while only 25% of the manures were treated in manure management system (FAOSTAT, 2019). The unattended manure has directly/indirectly led to the worsening of land, water, air and sight pollution. In fact, these manures possess the potential to be converted into valuable end-products (Janković, 2018) through different chemical conversion process. Traditionally, these manures are only used to fertilise crops or just being buried in landfills. However, due to the implementation of stricter environment regulation, there is an urgent need for different disposal ways in order to minimize land and water pollution (Ro et al., 2007).

HM is one of the many types of biomass waste sources with the potential to be converted into biofuels through thermochemical conversion process. Conventional methods in managing HM in Malaysia and other countries include composting for fertilizers, anaerobic digestion for biogas and incineration for power production. As a matter of fact, these methods have contributed to environmental pollutions in one way or another. Thus, alternative methods in effectively managing the HM are required. Interestingly, it has been reported in literature that animal manure is a potential candidate for biofuel production through thermochemical conversion process. To date, HM is seldom being studied as a thermal decomposition feedstock due to the lack of mass loss behaviour and kinetic properties.

Among the available thermal decomposition processes (eg: torrefaction, combustion, gasification) considered in valorising HM, pyrolysis is preferred as it could yield usable amount of end products in all three forms of solid, liquid and gas. Moreover, the process emits a lower amount of gaseous pollutants when compared with other thermal decomposition methods. The commonly adopted heating mechanisms for the pyrolysis process can be divided into two categories: 1. Conventional heating and 2. Microwave heating. Microwave heating is deemed to be more superior when compared with conventional methods as it transfers heat through radiative means by heating the feedstock internally, thus, promoting a more uniformed heating across the feedstock.

To date, it is also to note that a swine manure pyrolysis facility has been setup in Belgium with an annual operation capacity of 7,000 ton (Rajabi Hamedani et al., 2019). Even though biomass wastes are commonly assumed to be carbon neutral sources, the entire pyrolysis process on swine manure was interestingly reported to have a significant undesirable impact on the environment when evaluated through a Life Cycle Analysis. Therefore, it is essential to conduct proper environmental impact evaluation in assessing the overall environmental effects of the entire microwaveinduced pyrolysis process based on the usage of HM as the feedstock.

### **1.3** Problem Statement

The increase in worldwide energy demand has prompted the search for alternative green renewable fuel. Biofuel has been widely explored and used for energy production in order to cope with the increasing energy demand. However, the energy supply and demand gap are still large. Increasing the production of first-generation biofuel, which is mainly derived from food sources, such as animal fat and vegetable oil, could increase the competition between resources for food consumptions and biofuel production (Naik et al., 2010). Hence, it is only imperative that biofuel production emphasis is shifted towards second-generation biofuel manufactured from biomass in order to diversify the biofuel resources.

From the literature, the thermal decomposition of HM has not been explored extensively. This might probably be due to the lack of insight and understanding on the behaviour of HM during thermal decomposition. In view of the current HM managing methods (composition, anaerobic digestion and incineration), which is not environmentally sustainable, alternative processing route is required. Thermal decomposition using microwave pyrolysis is proposed in the present study to upgrade HM into higher value end products. In accordance to the focus of second generation biofuel on waste-to-energy concept, the present study aims to microwave-pyrolyse HM in producing useful alternative second-generation bio-fuel.

#### **1.4 Research Objectives**

In order to achieve the aim of the study, the objectives of the investigation are:

- (a) To determine the thermal decomposition kinetics and thermodynamic properties of HM using thermal-gravimetric analysis (TGA).
- (b) To derive a correlation between the end-product yield of thermal-decomposedHM and the process parameters using design of experiment (DOE) method.
- (c) To identify potential final usage of bio-products derived from the thermal decomposition of HM at different process parameters.
- (d) To evaluate the environmental impact of HM-based microwave pyrolysis plant in Peninsula Malaysia through cradle-to-gate Life Cycle Analysis.

#### **1.5 Research Questions**

The research questions to be answered during this study have been formulated as follow:

- (a) What is the thermal decomposition kinetics and thermodynamics properties of HM?
- (b) How does the thermal decomposition of HM process parameters correlate with the end-product composition?
- (c) What is the possible end usage of bio-products obtained from thermal decomposition of HM?
- (d) Is HM a feasible feedstock for pyrolysis in terms of economy, energy balance and environmental effects?

### 1.6 Research Scopes

The scope covered by the present study is listed below:

- (a) To determine the kinetics properties of HM during pyrolysis through thermal decomposition using a thermogravimetric analyser
- (b) To design and built a pyrolysis rig implementing microwave heating technique
- (c) To quantify end-products (solid, liquid and gas) of thermally decomposed HM at varying process parameters
- (d) To correlate thermal decomposition process parameters (temperature, AC/HM ratio and carrier gas flow rate) with the yield of end-products using DOE method
- (e) To propose possible applications based on the distinct properties of produced end-products

(f) To evaluate the economic, energy yield and environmental impact on process of microwave pyrolysis on HM

#### 1.7 Research Framework

The framework for the present study is presented in Figure 1.3. In this research, the kinetics and thermodynamic properties of HM is determined using TGA. A microwave rig, capable of thermal decomposition, is developed. The yield of end-products at different combinations of thermal decomposition process parameters will be recorded. A mathematical model, correlating thermal decomposition process parameters of thermally decomposed HM end-products, will be derived. The fuel-based properties of thermally decomposed HM end-products are then obtained. Through quantitative and qualitative approaches, relevant and suitable applications for these end-products will then be proposed, ensuring effective usage of the derived second-generation biofuel. Lastly, a life-cycle analysis has been conducted on a microwave pyrolysis plant situated in Peninsula Malaysia and its environmental impact is assessed and compared with existing pyrolysis plant.

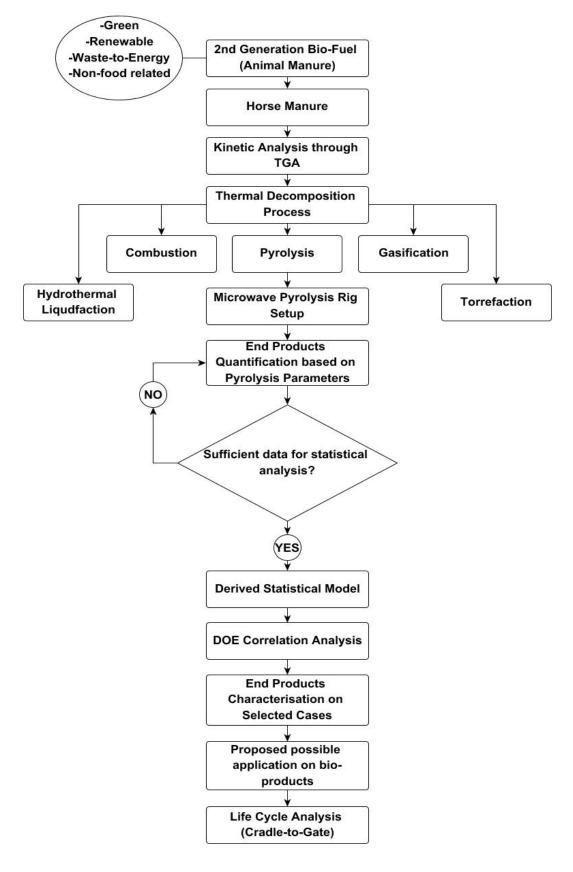


Figure 1.3 Research Framework

#### **1.8 Research Deliverables**

At the end of this study, it is expected that the following outcomes will be delivered:

- (a) The thermal decomposition kinetics and thermodynamic properties of HM will be determined.
- (b) A correlation between end-product yield and thermal decomposition process parameters will be established using design of experiment (DOE) method.
- (c) Possible end usage of bio-products derived from microwave pyrolysis of HM will be identified.
- (d) The environmental impacts of a microwave pyrolysis plant that process HM will be evaluated

From the derived correlation, it will then be possible to optimize the thermal decomposition process parameters for HM feedstock, depending on the desired end-product composition. A life cycle analysis will be conducted for HM using cradle-to-gate method and the feasibility in terms of economics, energy yield and environmental impacts will be presented. It is also noted that the derived framework can be adapted accordingly for thermal decomposition of various types of feedstock.

#### 1.9 Significance of Study

The study explores the thermal decomposition of HM and the potential use of its end products. The first part of the study attends to the need to evaluate the mass loss behavior and kinetics properties of HM, which has yet to be reported in literature. The discovered properties of HM under thermal decomposition can then be applied to deduce possible processing methods for HM. The subsequent part of the study implements microwave heating in pyrolysis, presenting the integration of nonconventional heating mechanism that improves overall heating efficiency for such a thermal conversion process. Through statistical analysis, a correlation between process parameters and product yield is derived, providing an essential fundamental foundation in the upsizing of pyrolysis reactor size. Such a correlation, coupled with fuel-based properties/characteristics, could prove to be essential in deciding on the potential usage of HM thermal decomposition end-products as a new and effective source of secondgeneration biofuel. The end products obtained in each phase (solid, liquid and gas) from pyrolysis of HM are also characterized to identify their respective potential end usages. Finally, the life-cycle analysis conducted in the last part of the study provides further evidence on the feasibility of HM-based microwave pyrolysis process when adopted at an industrial-scale. Overall, it is also to highlight that the entire study also presents a fundamental platform that can be duplicated to evaluate the valorisation of any other types of biomass waste.

#### 1.10 Summary

This chapter introduces the research problem, aims and objectives of the present study. The scope and significance of the study are also being highlighted. The following chapter covers the relevant literature review, corresponding to the research objectives identified in this chapter.

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- (a) Mong, G. R., Chong, C. T., Ng, J. H., Chong, W. W. F., Lam, S. S., Ong, H. C., Ani, F. N. (2020) Microwave Pyrolysis for Valorisation of Horse Manure Biowaste. Energy Conversion and Management 220, 113074. https://doi.org/10.1016/j.enconman.2020.113074. (Q1, IF:7.181)
- (b) Chiong, M. C., Valera-Medina, A., Chong, W. W. F., Chong, C. T., Mong, G. R., Mohd Jaafara, M. N. (2020) Effects of swirler vane angle on palm biodiesel/natural gas combustion in swirl-stabilised gas turbine combustor. Fuel 227, 118213. https://doi.org/10.1016/j.fuel.2020.118213. (Q1, IF:5.128)
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