OPTIMISATION OF PYROLYSIS OIL THROUGH MICROWAVE-INDUCED IN-SITU CO-PYROLYSIS OF WASTE TRUCK TIRE AND EMPTY FRUIT BUNCH

RUBIA BINTI IDRIS

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Faculty of Engineering
Universiti Teknologi Malaysia

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DEDICATION

I dedicate this to my beloved parents, my best friend, siblings and teachers without whom it was impossible for me to complete my thesis work.

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In the name of Allah, The Most Gracious and The Most Merciful. Alhamdulillah, praises to All Mighty Allah for the strengths and blessings in completing this thesis. Prayers and blessings on our beloved Prophet Muhammad صلى الله عليه وسلم

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ABSTRACT

Thermal decomposition of waste via pyrolysis is capable of producing pyrolysis oil. However, the produced oil tends to be unstable due to its poor physio-chemical properties such as high sulphur content, high acidic pH and high moisture content, and hence limiting its potential implementation as a fuel. Therefore, a microwave-induced *in-situ* catalytic fast co-pyrolysis study was proposed, serving to upgrade the pyrolysis oil to possess fuel-like properties. In the present study, co-pyrolysis of empty fruit bunch (EFB) with waste truck-tire (TT) were utilised with TT being selected because of its high volatile carbon content level and high heating value. Carbonaceous susceptor was also used to elevate the pyrolysis temperature. Firstly, pyrolysis temperature of microwave-induced fast pyrolysis of TT and EFB was optimised individually for increased pyrolysis oil yield and energy recovery. It was found that temperature of 500°C produced highest TT and EFB pyrolysis oil yields, which were approximately 38.12 and 38.26 wt%, respectively. TT pyrolysis oil was observed to consist of high calorific value (42.39 $MJkg^{-1}$) and high energy yield (40.55 wt%) but with high sulphur content. EFB was found to produce phenolic-rich pyrolysis oil with lower flash point, consisting of highly oxygenated compounds (90%) and high-water content (30 wt%). To overcome the lack of fuel-like properties, pyrolysis oil yield and energy recovery optimisation of co-pyrolysis between TT and EFB were conducted using responses surface methodology (RSM). Three parameters were examined, namely: 1) EFB to TT ratio, 2) pyrolysis temperature and 3) carbonaceous susceptor loading. It was observed that optimum conditions of 505°C pyrolysis temperature, 65% of EFB to TT ratio and 60g of susceptor loading produces highest pyrolysis oil yield (39.87 wt%) and energy recovery (60%). Such a co-pyrolysis configuration produced olefin correlate rich pyrolysis oil (39%) with high selectivity of D-limonene (28.6%) and 20% higher energy recovery as compared to TT pyrolysis oil. However, the liquid-oil still has a significant number of sulphur (0.05%) and acidic compounds (0.83%), mainly originating from TT and EFB. Thus, a microwave-induced in-situ catalytic fast co-pyrolysis of TT with EFB, using catalysts, was been carried out. Two parameters were studied: 1) catalyst types, namely activated carbon (AC), clay (CL) and calcium oxide (CaO), and 2) catalyst loading (ranging from 20 to 60%). It was shown that catalytic cracking decreases acidity of pyrolysis oil from 4.70 (un-catalytic) to 5.12 (AC20), 4.98 (CL20) and 5.65 (CaO20). As compared to CL and CaO, catalytic cracking using AC increased desirable hydrocarbon fractions (olefinic and monoaromatic) with highest selectivity of benzene, toluene, ethylbenzene and xylene (BTEX) hydrocarbons, indicating that such a catalytic cracking favours production of pyrolysis oil with fuel-like properties. It is, thus, parametrically determined that at 500°C pyrolysis temperature with 65:35 ratios (EFB/TT), 60g of susceptor, 20% of catalyst loading using AC at reaction time of 30 minutes, pyrolysis oil with highest yield of 38.92 wt% as well as highest energy recovery of 60.77% can be produced. The physiochemical properties of the pyrolysis oil were also determined to be similar to that of petroleum diesel but with a slightly lower flashpoint (<30°C). Thus, this work successfully demonstrated that microwave-induced catalytic co-pyrolysis of TT/EFB, using AC as catalyst, is a promising technique to recover diesel-like fuel from waste feedstocks, carrying great potential for use as supplemental alternative fuel or for value-added petrochemical products recovery.

ABSTRAK

Penguraian haba bagi sisa bahan terbuang melalui pirolisis mampu menghasilkan minyak pirolisis. Walau bagaimanapun, minyak yang terhasil tidak stabil kerana mempunyai sifat fisiokimia yang rendah seperti kandungan sulfur tinggi, pH berasid dan kandungan lembapan tinggi, menghadkan potensinya sebagai bahan bakar. Oleh itu, kajian ko-pirolisis cepat bermangkin menggunakan gelombang-mikro telah dicadangkan untuk meningkatkan kualiti minyak pirolisis bersifat bahan bakar. Dalam kajian ini, ko-pirolisis tandan buah kosong (EFB) dengan trak tayar terpakai (TT) digunakan kerana TT mempunyai kandungan karbon terurai dan nilai pemanasan yang tinggi. Penyerap karbon juga digunakan bagi meningkatkan suhu pirolisis. Pada awalnya, suhu pirolisis cepat gelombang-mikro TT dan EFB dioptimumkan secara individu bagi meningkatkan hasil minyak pirolisis dan tenaga dipulihkan. Didapati suhu 500°C menghasilkan minyak pirolisis TT dan EFB yang tinggi iaitu 38.12 dan 38.26 wt%. Minyak pirolisis TT diperhatikan mempunyai kalori (42.39 $\mathrm{MJkg^{-1}}$) dan tenaga (40.55 wt%) yang tinggi tetapi mengandungi sulfur yang tinggi. Minyak pirolisis EFB juga didapati dapat menghasilkan minyak pirolisis kaya-fenolik dengan takat kilat yang rendah, mengandungi sebatian beroksigen (90%) dan kandungan air (30 wt%) yang tinggi. Bagi mengatasi kelemahan sifat bahan bakar yang rendah, pengoptimuman hasil minyak dan tenaga terpulih ko-pirolisis antara TT dan EFB dilakukan dengan menggunakan *responses surface methodology* (RSM). Tiga parameter dikaji iaitu: 1) nisbah EFB ke TT, 2) suhu pirolisis dan 3) muatan karbon penjerap. Didapati keadaan optimum pada suhu ko-pirolisis 505°C, nisbah 65% EFB ke TT dan 60g muatan bahan penjerap menghasilkan minyak pirolisis (39.87 wt%) dan tenaga terpulih (60%) yang tinggi. Konfigurasi ko-pirolisis ini menghasilkan minyak pirolisis kaya olefin (39%) dengan keterpilihan tinggi D-limonene (28.6%) dan pemulihan tenaga 20% lebih tinggi berbanding minyak pirolisis TT. Walau bagaimanapun, minyak masih mempunyai kandungan sulfur (0.05%) dan sebatian berasid (0.83%), yang berasal dari TT dan EFB. Oleh itu, ko-pirolisis bermangkin TT dengan EFB menggunakan gelombang-mikro telah dilakukan. Dua parameter dikaji iaitu: 1) jenis mangkin iaitu karbon teraktif (AC), lempung (CL), kalsium oksida (CaO), dan 2) muatan mangkin (20 hingga 60%). Hasil kajian menunjukkan proses pemangkinan dapat menurunkan pH minyak pirolisis dari 4.70 (un-Cat) kepada 5.12 (AC20), 4.98 (CL20) dan 5.65 (CaO20). Berbanding dengan CL dan CaO, proses pemangkinan menggunakan AC meningkatkan pecahan hidrokarbon yang dikehendaki (olefinik dan monoaromatik) dengan keterpilihan tinggi hidrokarbon benzena, toluena, etilbenzena dan xilena (BTEX), menunjukkan proses pemangkinan menghasilkan minyak pirolisis bersifat bahan bakar baik. Oleh itu, parameter suhu pirolisis 500°C dengan nisbah 65:35 (EFB/TT), 60g muatan bahan penjerap dan 20% muatan mangkin AC pada masa tindakbalas 30 minit, mampu menghasilkan minyak pirolisis yang tinggi 38.92% serta tenaga terpulih yang tinggi 60.77%. Sifat fisiokimia minyak juga didapati setanding dengan petroleum diesel tetapi titik kilat rendah sedikit (<30°C). Oleh itu, kajian ini berjaya menunjukkan bahawa ko-pirolisis bermangkin TT/EFB dengan gelombang-mikro menggunakan AC sebagai mangkin adalah kaedah yang berpotensi dalam penghasilan semula bahan bakar mirip diesel dari bahan buangan sisa untuk digunakan sebagai bahan bakar alternatif tambahan atau untuk penghasilan semula produk petrokimia bernilai tambah.

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LIST OF ABBREVIATIONS

AC : Activated carbon
ANOVA : Analysis of variance
AR : Allylic radicals
ASTM : America standard testing
BET : Brunauer-Emmett-Teller surface area analyser
BFB : Bubbling Fluidized bed
BR : Butyl rubber
BT : Bicycle tire
BTEX : Benzene, toluene, ethylbenzene and xylene
CA : Continuous auger
CAC : Coconut activated carbon
CaO : Calcium oxide
CBS : Conical Spouted Bed
CCD : Central composite design
CFP : Catalytic fast pyrolysis
CHNSO : Carbon, hydrogen, nitrogen, oxygen and sulphur
CL : Clay
CPO : Crude palm oil
CR : Chloroprene rubber
CS : Cotton stalk
CV : Coefficient of variation
co-FP : Fast co-pyrolysis
co-CFP : Catalytic fast co-pyrolysis
D : Density
DF : Degree of freedom
DTG : Differential Thermo Gravimetry
E : Energy
EA : Elemental analysis
EFB : Empty fruit bunch
EFB100 : Empty fruit bunch pyrolysis oil 100%

ELTs : End-of-life tires
EPO : EFB pyrolysis oil
FB : Fixed Bed
FC : Fixed carbon
FESEM : Field-emission scanning electron microscope
FGV : Felda Global Venture
FP : Fast pyrolysis
FTIR : Fourier-transform infrared spectroscopy
GCMS : Gas chromatography spectroscopy
GHGs : Greenhouse gasses
HHV : Higher heating value
HR : Heating rate
KV : Kinematic viscosity
LHV : Lower heating value
MAHs : Monoaromatic hydrocarbons
MF : Mesocarp fruit fibre
MFS : Mixed feedstock
MIP : Microwave-induced Pyrolysis
MPOB : Malaysia Palm Oil Board
MW : Microwave
NaCl : Sodium Chloride
NR : Natural rubber
OPW : Oil palm waste
OPT : Oil palm trunk
PAHS : Polyaromatic hydrocarbons
PBR : Polybutadiene rubber
PCT : Personal car tire
PKS : Palm kernel shells
PO : Pyrolysis oil
PO65 : Pyrolysis oil ratio 65:35 EFB/TT
POME : Palm oil mill effluent
PP : Pour point
RO : Rotary oven
RS : Rice straw

RSM	:	Response surface methodology
SBR	:	Styrene–butadiene copolymer
SCB	:	Sugarcane bagasse
SD	:	Standard deviation
SiC	:	Silica carbide
SPT	:	Soapstock
SR	:	Synthetic rubber
SS	:	Sum of squares
ST	:	Scrap tire
TGA	:	Thermogravimetric Analysis
TDF	:	Tire-derived-fuel
TPO	:	Tire pyrolysis oil
TTPO	:	Truck-tire pyrolysis oil
TT	:	Truck-tire
TT100	:	Truck tire pyrolysis oil 100%
UT	:	Used tire
VM	:	Volatile matter
WT	:	Waste tire

LIST OF SYMBOLS

A - Amperes

CH_3COOH - Acetic acid

cm - Centimetre

CO - Carbon monoxide

CO_2 - Carbon dioxide

^{o}C - Degree Celsius

$^{o}C.min^{-1}$ - Degree Celsius per minute

Q - Heat of water

g - Grams

GHz - Gigahertz

$J.^{o}C^{-1}$ - Joules per degree Celsius

Kw - Kilowatt

λ - Lamda

L - Liter

m - Meter

$MJkg^{-1}$ - Mega joule per kilogram

min - Minute

μL - Micro Liter

μm - Micrometer

mm - Millimeter

$mL.min^{-1}$ - Milliliter per minute

μA - Micro Amperes

$Mg.L^{-1}$ - Milligram per Liter

MPa - Mega Pascal

MHz - Megahertz

n - Number of experiments

N - Number of factors

n_A - Number of axial points

n_F - Number of factorial points

D_p - Penetration depth

H_2O_2 - Peroxide acid
R - Regression coefficient
Re - Reynold Number
s - Second
H_2SO_4 Sulphuric acid
t - Slope of the calibration curve
$\tan \delta$ - Tangent alpha
% - Percent
W - Watts

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The increasing energy demand in the transportation sector along with the need to reduce CO_2 emission have prompted the search for innovative technology and renewable energy sources. Although there are numerous alternative energy sources that could potentially substitute fossil fuels, critical factors, such as availability of the fuel source, economic viability and environmental impacts remain the biggest challenges when considering fuel candidates. For transportation purposes, liquid fuel still appears as the most attractive and feasible form of fuel when taking into account energy density, stability and existing infrastructure (Liu *et al.*, 2014). In recent decades, much research has been devoted to produce alternative liquid fuel from solid municipal, industrial and agricultural wastes (Suriapparao *et al.*, 2018; Li *et al.*, 2019; Wang *et al.*, 2020). Aside from contributing towards value-added chemicals production, waste recycling also presents itself as a viable form of renewable energy, potentially leading to direct benefits to the environment and the economy (Ding *et al.*, 2019; Wang *et al.*, 2019).

When considering waste-to-energy recycling, waste tire presents itself as one of the potential sources of solid waste. The growing number of vehicles on the road worldwide generates millions of used tires annually. Improper management of waste tires has thus far created a huge environmental problem. For example, due to the artificial and non-biodegradable polymer used in the production of these tires, waste tires become difficult to decompose under natural environment. Alternatively, this led to improper incinerations, which further release hazardous pollutants (including polycyclic aromatic hydrocarbons (PAHs), benzene, styrene, phenols, and butadiene) to the atmosphere that could severely affect human health (Hita *et al.*, 2016; Idris *et al.*, 2019). Aside from this, landfilling of waste tire could also pose as a serious fire hazard. The uncontrolled combustion of such waste tires results in the release of black smoke in an enormous volume. The released sulphur content and additives from this combustion could pollute the environment (Pilusa, 2017; Dai *et al.*, 2018).

In order to overcome the potential environmental harm done due to improper management, waste tires have been often considered to be recycled for energy recovery (Ismail *et al.*, 2017; Gamboa *et al.*, 2020). However, to date, drawbacks of processing waste tire and high operating cost remain issues that have to be properly tackled. Recently, the urgent need to dispose waste tire efficiently in the context of circular economy has further provided the impetus for researchers to further explore thermal conversion process, via pyrolysis, on waste tire (Idris *et al.*, 2019; Mkhize *et al.*, 2016; Rodríguez *et al.*, 2020). Generally, pyrolysis involves thermal decomposition of materials at elevated temperatures (300 to 700 °C), in an oxygen-free environment, to decompose solid wastes into biochar, pyrolysis oil and syngas. Quality and yields of the pyrolysis oil are often strongly dependent on the operating conditions, such as particle size, temperature, heating rate, reaction atmosphere and type of reactor as well as the properties of the feedstock (Kabir and Hameed, 2017; Zhang *et al.*, 2017; Uzoejinwa *et al.*, 2018). The low heat transfer and high residence times parameters are significantly influence the biochar quality (Brassard *et al.*, 2017; Li *et al.*, 2019). While, the type of reactor, catalyst, reaction temperature, carrier gas and residence time plays an important role in producing high quality syngas (Policella *et al.*, 2019; Lin *et al.*, 2020). In this study the upgrading of pyrolysis oil properties nearly similar to transport-grade diesel or gasoline has been investigated due to the waste tire pyrolysis oil has high calorific value thus, potential use as a drop-in fuel in existing engine (Gamboa *et al.*, 2020).

The waste tire is a polymeric compound (consisting of natural and synthetic rubber) and is expected to play a major role in affecting the pyrolysis oil composition. The composition of the pyrolysis oil depends on the type of tire, i.e. personal car tire (PCT) or truck-tire (TT). As an example, TT has been known to have larger natural rubber content when compared with PCT. On the other hand, synthetic rubbers, such as butadiene and styrene butadiene, represent a third of the rubber in TT and two-thirds in PCT, respectively. Sienkiewicz et al. (2012) reported that, based on US and EU standards, natural rubber content in PCT and TT accounts for 14 to 22 wt% and 27 to 30 wt%, respectively. The synthetic rubber in PCT is found to be 27 to 30 wt% while for TT, it is approximately 14 to 15 wt% (Sienkiewicz *et al.*, 2012; Efika *et al.*, 2018). Therefore, it is expected that the difference in polymeric composition in waste tire results in different thermal degradation characteristics. Subsequently, the quality of pyrolysis oil produced also vary (Ozcan *et al.*, 2016; Kan *et al.*, 2017; Han *et al.*, 2018).

In exploring thermal conversion of different polymeric compounds, Seidelt et al. (2006) examined the thermal properties of natural and synthetic rubber, which are derived from styrene–butadiene-rubber and polybutadiene rubber, respectively. Their results revealed that there exists a relationship between the polymeric composition and thermal degradation of three different rubbers, thus, giving different pyrolysis oil compositions (Bockhorn *et al.*, 2006). Pyrolysis of waste tire also allows for the degradation of polymeric compounds into lower molecular weight oil that could be used as alternative fuels or chemicals feedstock (Undri *et al.*, 2013; Alvarez, *et al.*, 2017). The pyrolysis oil has been reported in literature to have high calorific values (40 to 45 $\mathrm{MJkg^{-1}}$), which typically consists of a mixture of aliphatic, olefinic and aromatic hydrocarbons, depending on the process conditions and tire composition (Alvarez, *et al.*, 2017; Idris *et al.*, 2019). However, many studies also reported that the pyrolysis oil contains a significant amount of nitrogen and sulphur compounds, making this oil inferior as compared to fossil fuel. Consequently, this prohibits direct usage of such oil in an engine.

Additionally, the produced pyrolysis oil has also been characterized to have poor physical properties, such as high viscosity, low flash point and low density that affects the spray injection system (Song *et al.*, 2018; Suntivarakorn *et al.*, 2018). Das et al. (2018) reported that high viscosity and low volatility of liquid fuel tend to result in inferior atomization and reduced fuel vaporization, whereas high fuel density leads to the increase in spray penetration that results in the increase in emissions of unburnt hydrocarbon and carbon monoxide, CO (Das *et al.*, 2018). High water content in the oil also affects the energy content (Das *et al.*, 2018) while high sulphur content could corrode the internal components of the engine, such as piston ring, valves and cylinder liners (Suntivarakorn *et al.*, 2018).

Thus, it is imperative that the pyrolysis oil quality be improved before being considered for direct application in existing engines. Recently, co-pyrolysis and catalytic co-pyrolysis processes have been much adopted in the process to improve the quality and the quantity of pyrolysis oils. Several studies have reported improved pyrolysis oil properties and yield through co-pyrolysis and catalytic co-pyrolysis, suggesting that the synergistic effects between two different materials during the pyrolysis process could lead to positive effects (Shah *et al.*, 2019; Fakayode *et al.*, 2020). Previous literature shows that catalytic co-pyrolysis studies have been performed mostly by using a fixed-bed reactor, aiming to improve liquid oil properties and yield. As an example, Zeeshan et al. (2018) conducted a study on co-pyrolysis of the waste tire (WT) and sugarcane bagasse (SCB) under a fixed-bed reactor. From their study, it revealed the optimised ratio of WT/SCB at 1:3 produced the highest yield of pyrolysis oil (49.7 wt%) as compared to SCB single feedstock (42.1 wt%). Adding WT to SCB have been observed to significantly increase the calorific value of oil from 19.1 $MJkg^{-1}$ (SCB) to 41.0 $MJkg^{-1}$ (WT/SCB; 1:3) (Ahmed *et al.*, 2018). Shah et al. (2019) also studied the co-pyrolysis of the cotton stalk (CS) with waste tire (WT) also under a fixed bed reactor. It showns that adding WT to the CS pyrolysis feedstock resulted in improved oil yield and quality. The optimised ratio of CS/WT achieved at 2:3 resulted in maximum oil yield of 48.0 wt% and a high organic phase of 78.0 wt%. The research showed that adding WT to CS resulted in a significant increase in carbon content and a decrease in the oxygen content of the pyrolysis oil, while the calorific value improved from 23.6 $MJkg^{-1}$ (pure CS) to 41.3 $MJkg^{-1}$ (CS/WT ratio, 2:3) (Shah *et al.*, 2019).

Likewise, microwave heating pyrolysis has also recently been shown as a promising route for waste feedstock recycling into renewable fuel and value-added materials (Ali and Idris, 2016; Beneroso *et al.*, 2017; Suriapparao *et al.*, 2018). The use of microwave heating is reported to provide various advantages with respect to rapid heating, volumetric heating, selective heating and short processing time as compared to conventional heating (Bhattacharya and Basak, 2016; Antunes *et al.*, 2018; Fan *et al.*, 2019). Dai et al. (2017) conducted a study of microwave-assisted catalytic fast co-pyrolysis of soapstock (SPT) with waste tire (WT) using HZSM-5 catalyst. The optimal co-pyrolysis condition, at 550 °C with the catalyst-to-feedstock ratio of 1:5, showns to result in the highest yield of hydrocarbons fraction. The use of catalyst was also observed to have enhanced the total amount of olefins and aromatics produced between 82.1 to 89.4 % but with reduced the yield of pyrolysis oil from 42.0 wt% (catalyst-free) to 38.9 wt% (with-catalyst) (Dai *et al.*, 2017). From their study, it revealed that WT demonstrated a significant synergistic effect with SPT in facilitating the production of hydrocarbon and aromatics compounds in the pyrolysis oil (Dai *et al.*, 2018).

Wang et al. (2018) also studied the microwave-assisted catalytic co-pyrolysis of soybean straw (SS) and soapstock (SPT) using SiC ceramic as a catalyst. It was reported that the pyrolysis oil yield decreased from 49.8 wt% (catalyst-free) to 41.3 wt% (with-catalyst). However, the ratio of oxygen-containing compounds decreased from 34.0% (catalyst-free) to 23.0%. From that point of view, the catalytic co-pyrolysis process showed a synergistic reaction catalysed by SiC to promote deoxygenation of oxygenated compounds. Moreover, microwave heating system has been observes to enhance the reaction rates while improving the pyrolysis oil properties (Beneroso *et al.*, 2015; Mutsengerere *et al.*, 2019).

From the literature, microwave heating has been demonstrated to be able to provide a slow but high heating rate due to the fact that microwave energy is delivered directly into the material through molecular interaction via the electromagnetic field with little wastage to the surrounding area. Significant savings of time and energy were achieved in the microwave-induced co-pyrolysis work conducted by Dai et al. (2018) and Wang et al. (2018). Higher heating rate improves the devolatilization of the feedstock by reducing the conversion time. Furthermore, the heating rate influences the residence time of volatiles that flows from the internal heating zones towards the external cold regions of the sample. The higher heating rate, shorter residence time and high volatilization rate reduce the activity of secondary reactions of vapor phase products. Consequently, this results in high yields of liquid and reduces the deposition of refractory condensable material on the char's internal surface (Asomaning *et al.*, 2018; State *et al.*, 2019).

Therefore, in this study, microwave-induced *in-situ* catalytic fast co-pyrolysis of waste truck tire with empty fruit bunch (EFB), involving three types of catalysts, namely activated carbon, clay and calcium oxide, were used to upgrade the quality of liquid oil as well as increased the energy recovery. The waste tire and empty fruit bunch (EFB) are considered as a suitable candidates for co-pyrolysis due to its high carbon content and heating value (Antoniou and Zabaniotou, 2015; Akkouche *et al.*, 2017). In view of the abundance of EFB and waste truck-tire, pyrolysing the materials present a viable route for energy recovery and waste reduction. Previous co-pyrolysis research on biomass with waste tire was mainly focused on the use of personal car tire (PCT) (Farooq *et al.*, 2017; Wang *et al.*, 2018; Alvarez *et al.*, 2019). However, the study of co-pyrolysis of EFB with waste truck-tire (TT), to the best of the authors' knowledge, has never been reported in literature. In this work, the microwave heating technique with the aid of carbonaceous susceptor is utilised to provide the heating source for pyrolysis.

Firstly, the optimisation temperature of microwave-induced fast pyrolysis TT and EFB individually. The effect of reaction temperature is studied to determine the liquid oil yield, chemicals composition, hydrocarbon fractions and higher heating value (HHV) as well as the energy yield of the liquid oil. Secondly, the optimisation of co-pyrolysis TT and EFB using responses surface methodology (RSM) is to be conducted. The central composite design (CCD) of RSM is utilised to optimise the experimental conditions of microwave-induced co-pyrolysis of TT/EFB. Three parameters are examined (EFB to TT ratio, reaction temperature and carbonaceous susceptor loading) to optimise the production of liquid oil and energy yield. The optimised results from the RSM study are conducted for the catalytic fast co-pyrolysis of waste truck tire with empty fruit bunch (EFB) using sustainable catalyst to further upgrade the pyrolysis oil properties. Two parameters are studied, which are the effect of catalyst (AC-activated carbon, CL-clay, CaO-calcium oxide) and the effect of catalyst loading (20 to 60%) to upgrade the quality of pyrolysis oil.

In conclusion, this work is expected to contribute to the database of waste truck tire and empty fruit bunch co-pyrolysis. Based on the concept of waste-to-energy approach, the findings of the present study are also expected to significantly contribute to the understanding of upgrading pyrolysis oil properties, increasing the liquid oil and energy yield as well as valuable chemicals recovery, such as phenolic, BTEX and limonene.

1.2 Problem Statement

Growing number of vehicles on the road worldwide has led to the generation of millions of end-life tires (ELTs) annually (Presti, 2013; ETRma, 2014). It was reported that about 1.4 billion of new tires are sold worldwide yearly and subsequently just as many falls into the category of end of life tires (ETRma, 2014). The Environmental Protection Agency reported that million tonnes of waste tires are generated yearly all over the world. These waste tires are among the most problematic sources of waste due to their nature of being non-decomposed and non-biodegradable. Waste tire accumulation pose a threat to public health, safety and the environment worldwide. The improper management of waste tires is creating a burden and environmental impact. Thus, several alternatives have been developed to manage waste tire via recycling and energy recovery. However, there are still drawbacks, such as high costs and high sulphur content of oil. Due to these drawbacks, many studies have been conducted on the waste tire co-pyrolysis with other materials, such as waste plastic, coal and sewage. Yet, little has been reported in literature in which the focus is on the co-pyrolysis of lignocellulosic biomass/waste tire, particularly with EFB biomass.

Likewise, the palm oil industry has brought a great economic benefit to Malaysia. However, abundance of waste biomass generated from the palm oil mills is significantly increasing annually (Loh *et al.*, 2017). The waste biomass includes trunks, fronds, empty fruit bunches and other biomass fractions. Malaysia Palm Oil Board, MPOB, (Board, 2017) reported that the total production of crude palm oil (CPO) in Malaysia was 3.4 million tonnes in 2016. Consequently, it is also reported that about 25.5 million tonnes of oil palm waste (OPW) were generated since 75 wt% of the solid wastes were produced from 10 wt% of CPO. Part of these OPW biomass flow is already being used for energy production, such as palm kernel shell (PKS) and empty fruit bunch (EFB) are utilised for palm mill boiler and remote local electric generator (MPOB, 2018). Additionally, EFB, OPF and OPT have also been utilised as mulching and fertilizer agent for the production of packaging and building materials. However, there are still a significant amount of waste biomass from the palm oil industry that have been left behind. These wastes can still be mobilized for improving waste biomass utilization efficiency in order to meet the current energy demand and to also sustain the palm oil industry (Liew *et al.*, 2018; Ahmad *et al.*, 2019)

Despite the fact that pyrolysis-oil is environmentally friendly, fuel characteristic of this oil remains undesirable as compared to fossil fuel, especially with regards to its combustion efficiency. Direct use of pyrolysis-oil is difficult due to its complex compositions, which contains acids and high level of oxygenated compounds. This characteristics have led to poor liquid fuel characteristic, such as high viscosity, corrosiveness, low heat value and instability of oil (Umeki *et al.*, 2016; Mutsengerere *et al.*, 2019). Several researchers have reported that oil from the pyrolysis process of waste tire and biomass alone generally consists of high sulphur, nitrogen oxygen in form of phenolic compounds with high water content (Chang, 2014; Guo *et al.*, 2017; Kim *et al.*, 2019) . Thus, it is necessary to improve the pyrolysis oil quality in order to overcome the challenges for it to be directly used for fuel-related applications.

It is clear that since pyrolysis process produces long chain hydrocarbon as a main product, the process requires breaking of carbon–carbon bonds to produce light hydrocarbon and aromatic compounds. This process can be catalysed by solid acid catalysts, such as zeolites, which are used in conventional petroleum oil refineries for the same purpose (Xie *et al.*, 2018; Wang *et al.*, 2019). In general, zeolite catalyst has been used as the main catalyst for pyrolysis of many different feedstocks to upgrade the pyrolysis oil properties. Previous studies showed that the utilization of zeolite catalyst in the biomass pyrolysis has dramatically changed the composition of pyrolysis oils by reducing the amounts of oxygenated compounds in pyrolysis oil via deoxygenation reactions. Simultaneously, this also resulted in the increase in aromatic compounds, producing a lighter fraction (gasoline-type fuel). A decrease in the pyrolysis oil molecular weight is hence obtained. However, zeolite is considered as an expensive catalyst (Zhao *et al.*, 2018; Sun *et al.*, 2019). This is due to the coke formation during the process that requires replacing of a fresh catalyst for every new cycle reaction. Thus, replacing zeolite catalyst with a more economised catalyst, such as activated carbon, calcium oxide and kaolin clay, are expected to lead to a more cost-effective catalytic pyrolysis process.

1.3 Motivation of the Study

Co-pyrolysis presents a viable method to upgrade the pyrolysis oil properties using the microwave-assisted heating method. This process involves two or more different materials being used as a feedstock. Many studies showed that co-pyrolysis using two different feedstocks, such as synthetic polymer and biomass, exhibited a synergistic effect during the pyrolysis process. It has also been observed that the co-pyrolysis also facilitates the production of light aromatics hydrocarbon, such as benzene, toluene, ethylbenzene and xylene (BTEX). The produced aromatic hydrocarbon is found to be a gasoline-type fuel hydrocarbon. Thus, the selection of feedstock presents itself as one of the crucial factors to improve the oil quality and quantity during the co-pyrolysis (Hassan *et al.*, 2016; Zhu *et al.*, 2018; Gu *et al.*, 2020). Abnisa and Daud (2015) reported that the synergetic effect of co-pyrolysis depends on the type and the contact of different materials, pyrolysis duration, temperature and heating rate, removal or equilibrium of volatiles formed and addition of solvents, catalysts along with hydrogen-donors (Abnisa and Daud, 2015).

The type of blending feedstock also has a significant influence as compared to the abovementioned other factors. Thus, synergistic effects on co-pyrolysis can be complicatedly varied based on the feedstock (Abnisa and Daud, 2015). In this study, adding waste truck tire (TT) into the empty fruit bunch (EFB) feedstock is expected to exhibit good synergetic effect, which is attributed to the higher fixed carbon and lower volatile matter of TT as compared to EFB (through proximate analysis study). Moreover, TT is considered as a good microwave absorber due to the content of carbon black, which complements the poor dielectric property of EFB. Thus, the addition of TT acts as the microwave absorber in enhancing the heating of EFB through heat conduction in facilitating the microwave heating process (Mushtaq *et al.*, 2015; Lam *et al.*, 2019). Generally, mixing of feedstock, under a co-pyrolysis process, is expected to result in different physicochemical properties that will invariably lead to a synergetic effect between the two materials, giving rise to end products of different quality and yield.

The utilisation of microwave heating pyrolysis has also been reported to provide various advantages with respect to rapid heating, volumetric heating, selective heating and short processing time as compared to conventional heating (Bhattacharya and Basak, 2016; Antunes *et al.*, 2018; Fan *et al.*, 2019). Numerous studies have been published on the efficiency of microwave pyrolysis using different materials (Song *et al.*, 2017; Asomaning *et al.*, 2018; Guedes *et al.*, 2018). The microwave can heat objects uniformly in a shorter heating time as compared to the conventional heating (Zhang *et al.*, 2018; Haeldermans *et al.*, 2019). In addition to this, activated carbon will also act as a good microwave susceptor to absorb microwave energy and converting into heat to be transferred to nearby particles of the feedstock through convection, conduction and radiation process (Bhattacharya and Basak, 2016; Antunes *et al.*, 2017). This significantly decreases the residence time of volatiles vapor in the hot zone of reactor vessel. Thus, the increasing of liquid oil yield as well as upgrading of the pyrolysis oil quality is to be expected. This is because rapid microwave pyrolysis heating reduces the probability of secondary cracking of volatile vapour during the pyrolysis process. As a result of this, the pyrolysis oil produced consists of relatively high proportion of olefins hydrocarbons, monoaromatics hydrocarbon, mainly from BTEX hydrocarbons (benzene, toluene, ethylbenzene and xylene) as well as high proportion of valuable chemicals, such as ethylene, propylene, butene and limonene (Uzoejinwa *et al.*, 2018; Martínez *et al.*, 2019; Idris *et al.*, 2020). The shorter processing time also contributes to the reduction in operating cost. Moreover, the pre-treatment of the feedstock, such as drying, grinding into a small particle size, acid or base treatment are the requirement in a microwave pyrolysis as compared to the conventional heating. Thus, the pre-processing cost of pyrolysis process can be significantly reduced from the economic point of view.

Catalytic co-pyrolysis also offers a facile method by implementation of catalyst into the fast co-pyrolysis process to upgrade the pyrolysis oil properties via cracking of heavy hydrocarbon into short chain hydrocarbons. Pattiya (2018) reported that the main reactions involved in the catalytic cracking are carbon-carbon bonds cleavage, isomerisation, polymerisation, condensation, alkylation and aromatisation. These reactions lead to the production of light hydrocarbons, such as aliphatic, olefins and monoaromatic hydrocarbons, BTEX (Pattiya, 2018). Many studies have used zeolite catalyst in achieving a more efficient bio-oil upgrading (Hu *et al.*, 2017; Zhao *et al.*, 2018; Zhou *et al.*, 2019). However, zeolite catalyst suffers from deactivation of coke formation and requires replacing of fresh catalyst for every new cycle. This is very ineffective from an economic point of view. Previous studies have showed that mirco-porous catalysts, such as activated carbon or biochar produced from the pyrolysis process, could potentially be used as a catalyst to upgrade liquid oil properties. This is due to their physio-co-chemical properties of activated carbon, such as high surface area, large pore volume and multiple pore size distribution (Miandad *et al.*, 2016; Dong *et al.*, 2018; Zhang *et al.*, 2019). Due to the coke formation and requirement of replacing a fresh catalyst for every new cycle during co-pyrolysis, switching from zeolite catalyst to a more sustainable catalyst, such as activated carbon or waste-derived activated carbon (biochar), can be expected to significantly reduce the operation cost for potential fuel production. This is due to the fact that activated carbon or waste-derived activated carbon (biochar) catalyst is in abundance and much cheaper as compared to zeolite catalyst.

1.4 Research Objectives

The aim of the present study is to upgrade the quality and yield of pyrolysis-oil derived from catalytic co-pyrolysis between waste truck tire (TT) and palm-based empty fruit bunch (EFB). To achieve the aim, the objectives of these study are presented as follow:

(a) To determine the properties of the pyrolysis oil derived from microwave-induced pyrolysis of TT.

(b) To determine the properties of pyrolysis oil derived from microwave-induced pyrolysis of EFB.

(c) To optimise the yield and energy recovery of pyrolysis oil derived from microwave-induced co-pyrolysis of TT and EFB.

(d) To upgrade the quality of pyrolysis oil derived from microwave-induced catalytic co-pyrolysis of TT and EFB using activated carbon, clay and calcium oxide catalysts.

By the end of the study, it is aimed that a satisfactory upgradation of the pyrolysis oil, derived through catalytic co-pyrolysis between TT and EFB, into potentially diesel-like fuel be achieved. The findings of the study are also expected as the strong fundamental platform to more effectively valorise wastes, such as TT and EFB.

1.5 Scope of the Study

The scopes of this study consist of four main parts, which are: i) materials preparation and reactor setup; ii) microwave-induced pyrolysis waste truck tire (TT) and empty fruit bunch (EFB) individually; iii) microwave-induced co-pyrolysis of TT with EFB using the RSM software; and lastly iv) microwave-induced *in-situ* catalytic fast co-pyrolysis of TT with EFB using catalysts. The stainless steel-free truck tire had been supplied from Eco Power Synergy Sdn Bhd, located in Klang, Selangor Malaysia. The EFB pellet, on the other hand, had been supplied from FGV Semanchu Palm Oil Mill, located in Kota Tinggi, Johor Bahru, Malaysia. The catalysts used in this study were commercial grade activated carbon, clay and synthesis calcium oxide. Details of the samples and the catalyst preparation are explained in Chapter 3. It is to note that the raw material and the catalysts have been characterized using several spectroscopic tecneques, such as GCMS, elemental analysis, bomb calorimeter, FTIR, XRD, TGA, FESEM and XRF, to evaluate the pyrolysis oil yield along with its chemical properties and energy recovery yield.

1.6 Summary

In this chapter, researched background discussed in brief the potential used of waste truck tire (TT) and empty fruit bunch (EFB) of palm-based biomass for the producing of pyrolysis oil. Due to low quality of pyrolysis oil produced from TT and EFB individually, the upgrading of pyrolysis oil is necessary for the direct application in diesel engine. Thus, the utilization of catalyst and microwave also has been highlighted for the upgrading of pyrolysis oil via microwave-induced *in-situ* catalytic co-pyrolysis TT with EFB for the potential application of drop-in fuel in engine. Many parameters have been investigated such as pyrolysis temperature, ratio of TT to EFB, weight loading of microwave susceptor, type of catalyst and catalyst-to-feedstock ratio. The quality of pyrolysis oil has been characterized using GCMS, elemental analysis, bomb calorimetry and general physiochemical properties such as viscosity, density, pH, flash point and moisture content.

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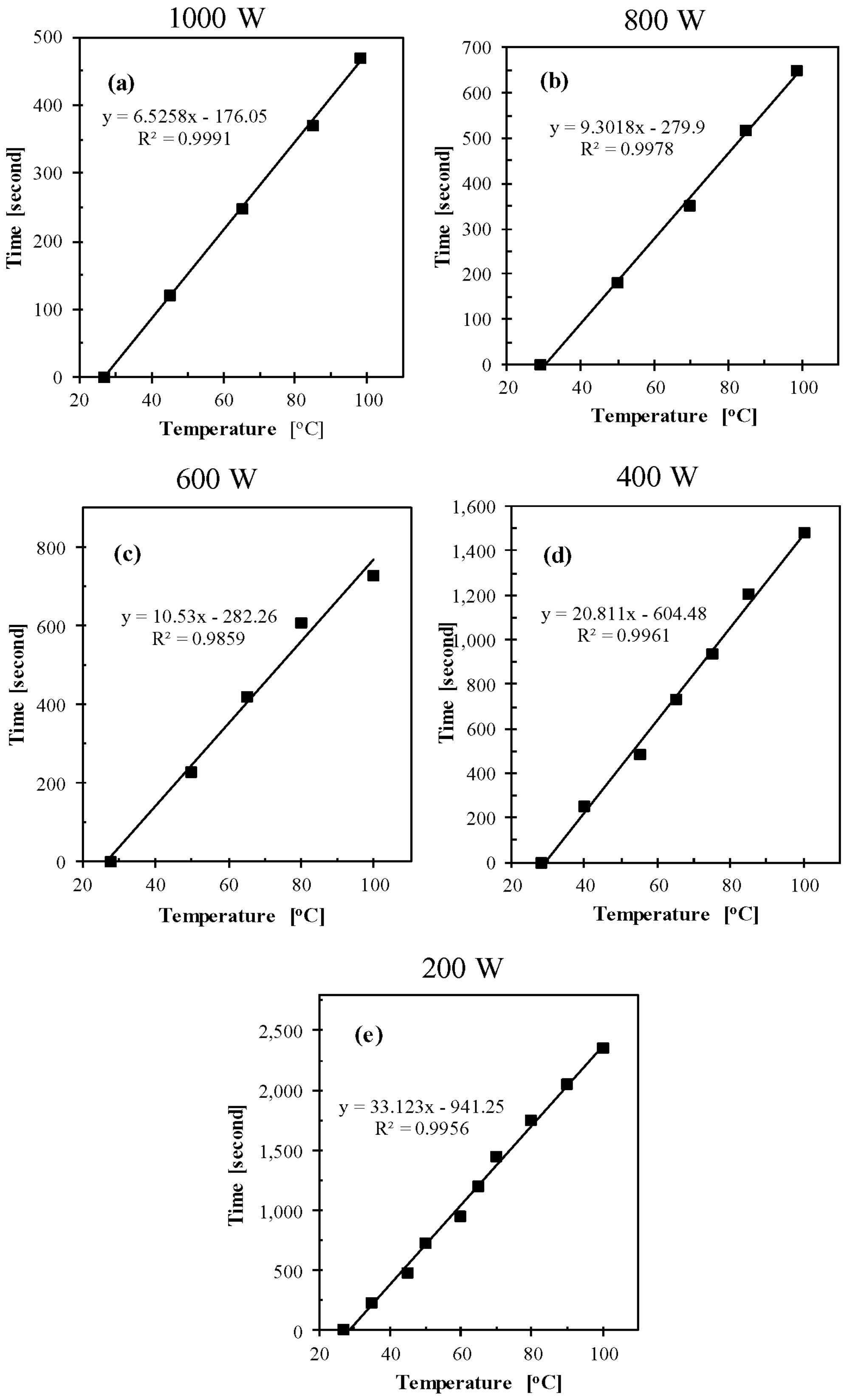
Appendix A Microwave power calibration graphs



SFig. 1. Microwave power calibration using 1000 g of distilled water at (a) 1000 W, (b) 800 W, (c) 600 W, (d) 400W and (e) 200 W

LIST OF PUBLICATIONS

Journal Papers

Idris, R., Chong, W.W.F., Ali, A., Idris, S., Hasan, M.F., Ani, F.N., Chong, C.T., (2021). 'Phenolic-rich Bio-Oil Derivation via Microwave-Induced Fast Pyrolysis of Empty Fruit Bunch with Activated Carbon as Microwave Susceptor', *Environmental Technology & Innovation*, 21, 101291.

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Idris, R., Chong, C. T., & Ani, F. N. (2019). Microwave-induced pyrolysis of waste truck tyres with carbonaceous susceptor for the production of diesel-like fuel. *Journal of the Energy Institute*, 92(6), 1831-1841.

Conference

Idris, R., Chong, W. W. F., Ali, A., Idris, S., Asik, J. A., Hasan, M. F., Ani, F. N. Effect of microwave susceptor design on the heating profile of co-pyrolysis between empty fruit bunches and waste truck tire. International Conference on Sustainable Energy and Green Technology (2019), 11-14 December 2019, Bangkok, Thailand.