MICROWAVE-ASSISTED PYROLYSIS AND CO-PYROLYSIS OF COAL AND OIL PALM SHELL WITH COCONUT SHELL ACTIVATED CARBON AS MICROWAVE ABSORBER

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Dedicated to

my beloved father, mother

and wife

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ABSTRACT

The increased energy insecurity and carbon dioxide (CO_2) emissions from fossil fuel utilization demands sustainable and cleaner fuel resources. Bio-fuel and chemicals from biomass have been recognized as renewable energy resource. Coal has the potential to become an important source for liquid and gas fuels. Co-processing of coal with biomass is considered a step towards sustainable and clean coal utilization. In this research work, oil palm shell (OPS) and coal were subjected to microwave (MW) pyrolysis and co-pyrolysis conditions to produce liquid fuels. Coconut activated carbon (CAC) used as a MW absorber was distributed uniformly over pyrolysis material to reduce hotspots. Three process parameters; CAC loading, MW power and nitrogen (N_2) flow rate were studied on pyrolysis performance. Initially, pyrolysis performance with 1, 2 and 3-Layer of carbons over isolated fuels were studied. Later, 3-Layer of carbon over isolated fuels was carried out with 35, 55 and 75 wt% CAC loading, increasing MW power and N_2 flow rate. The MW co-pyrolysis of coal and OPS in segregation and blend were investigated to observe vapor-phase synergy. The effects of process parameters on the efficiency of co-pyrolysis of blend were tested to identify the optimal processing conditions. The highest bio-oil and coal-tar of 36.26 wt% and 18.59 wt% were obtained with 75 wt% CAC loading using 450 W and 600 W with 4 liters per minute (LPM) of N_2 flow rate, respectively. This improved oil recovery is mainly due to the fact that higher MW power and CAC loading produced sustained pyrolysis conditions for longer duration for the complete conversion of fuel solids. The bio-oil was enriched in phenol with highest detected 71.77% gas chromatography-mass spectrometer (GC-MS) area with 3-Layer method at 75 wt% CAC loading, 300 W and 4 LPM of N_2 flow rate. This higher phenol formation can be attributed to the slow and uniform process heating conditions, and in-situ upgrading of pyrolysis vapors over successive carbon surfaces. The coal-tar is composed mainly of aromatics (naphthalenes, benzenes and xylene) and saturated aliphatics (alkanes and alkenes) hydrocarbons. The gas produced from pyrolysis of OPS and coal is H_2 with composition of 27.94–50.46 vol% and 40.23–65.22 vol%, respectively. The co-pyrolysis oil is composed of polars (phenol, phenolics and guaiacols) consisting of more than 50% GC-MS area. The MW co-pyrolysis in segregation of upper-bed-coal/bottom-bed-OPS produced higher polars of 71.62–76.33% GC-MS area with much limited aromatics and saturated aliphatics of 2.41–8.43% and 0.37–0.80% GC-MS area, respectively. Conversely, upper-bed-OPS/bottom-bed-coal segregated fuels produced lower polars of 50.92–61.82% GC-MS area with much higher aromatics and saturated aliphatics of 19.72–28.29% and 8.22–21.36% GC-MS area, respectively. The difference in polar, aromatics and saturated aliphatics in co-pyrolysis oil shows positive vapor-phase synergy. MW co-pyrolysis of blend optimum process conditions for 33.17 wt% oil were found to be at 71.38 wt% CAC loading, 582 W and 3.5 LPM of N_2 flow rate.

ABSTRAK

Peningkatan ketidaktentuan tenaga dan pelepasan karbon dioksida (CO_2) daripada penggunaan bahan api fosil membawa kepada permintaan sumber bahan api yang mampan dan lebih bersih. Bahan api bio dan bahan kimia daripada biojisim telah diiktiraf sebagai sumber tenaga yang boleh diperbaharui. Arang batu berpotensi untuk menjadi sumber penting untuk bahan api cecair dan gas. Pemprosesan bersama arang batu dengan biojisim dianggap satu langkah ke arah penggunaan arang batu yang mampan dan bersih. Dalam kajian ini, pirolisis gelombang mikro (MW) dan pirolisis bersama tempurung kelapa sawit (OPS) dan arang batu dilakukan untuk menghasilkan bahan api cecair. Karbon teraktif kelapa (CAC) digunakan sebagai penyerap MW diagihkan secara seragam ke atas bahan pirolisis untuk mengurangkan tompok panas. Tiga parameter proses; muatan CAC, kuasa MW dan kadar aliran nitrogrn (N_2) telah dikaji kesannya terhadap prestasi pirolisis. Pada mulanya, prestasi pirolisis dengan 1, 2 dan 3-lapisan karbon ke atas bahan api telah dikaji. Kemudian, 3-lapisan karbon ke atas bahan api telah dijalankan dengan 35, 55 and 75 wt% muatan CAC, peningkatan kuasa MW dan kadar aliran N_2. Pirolisis MW bersama arang batu dan OPS secara berasingan dan bergabung telah dilakukan untuk melihat sinergi fasa wap. Kesan parameter proses terhadap kecekapan pirolisis bersama campuran telah dilakukan untuk mengenal pasti keadaan pemprosesan optimum. Minyak-bio dan arang batu tar yang paling tinggi ialah 36.26 wt% dan 18.59 wt% diperolehi dengan 75 wt% muatan CAC menggunakan 450 W dan 600 W masing-masing dengan 4 liter seminit (LPM) kadar aliran N_2. Perolehan minyak yang lebih baik adalah disebabkan oleh kuasa MW dan muatan CAC yang tinggi dapat mengekalkan keadaan pirolisis pada masa yang lama untuk penukaran lengkap bahan api pepejal. Minyak-bio kaya dengan fenol paling tinggi pada luas kawasan kromatografi sebanyak 71.77% jisim gas spektrometer (GC-MC) dengan kaedah 3-lapisan pada 75 wt% muatan CAC, 300 W dan 4 LPM kadar aliran N_2. Pembentukan fenol yang lebih tinggi ini boleh dikaitkan dengan keadaan proses pemanasan perlahan serta seragam, dan peningkatan gred wap pirolisis secara in situ di permukaan karbon secara berturut-turut. Tar arang batu terdiri terutamanya daripada hidrokarbon aromatik (naftalena, benzena dan xilena) dan alifatik tepu (alkana dan alkena). Gas utama yang terhasil dari pirolisis OPS dan arang batu ialah H_2 masing-masing dengan kandungan 27.94–50.46 vol% dan 40.23–65.22 vol%. Minyak yang terhasil daripada pirolisis bersama terdiri daripada polar (fenol, fenolik dan *guaiacols*) yang mewakili lebih daripada 50% luas kawasan GC-MS. Pirolisis MW bersama dengan pengasingan lapisan atas arang batu/lapisan bawah OPS menghasilkan polar lebih tinggi dengan 71.62–76.33% luas kawasan GC-MS dan aromatik dan alifatik tepu yang terhad masing-masing dengan 2.41–8.43% dan 0.37–0.80% luas kawasan GC-MS. Sebaliknya, lapisan atas OPS/lapisan bawah arang batu menghasilkan polar yang lebih rendah iaitu 50.92–61.82 % luas kawasan GC-MS dengan aromatik dan alifatik tepu yang lebih tinggi masing-masing dengan 19.72–28.29% dan 8.22–21.36% luas kawasan GC-MS. Perbezaan polar, aromatik dan alifatik tepu dalam minyak pirolisis bersama menunjukkan sinergi yang positif bagi fasa wap. Keadaan proses optimum bagi pirolisis MW bersama campuran menghasilkan minyak 33.17 wt% adalah pada 71.38 wt% muatan CAC, 582 W dan 3.5 LPM kadar aliran N_2.

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LIST OF ABBREVIATIONS

3D	-	3 Dimensional
AC	-	Activated carbon
ANOVA	-	Analysis of variance
Approx.	-	Approximate
AR	-	Analytical reagent
Avg.	-	Average
BP	-	British petroleum
BS	-	British standard
BTL	-	Biomass to liquid
Btu	-	British thermal units
CAC	-	Coconut activated carbon
CCC	-	Central composite circumscribed
CCI	-	Central composite inscribed
CCD	-	Central composite design
CCF	-	Central composite face centered
CFT	-	Coal fired technology
CPPs	-	Coal power plants
CPS	-	Conventional pyrolysis system
CTL	-	Coal to liquid
d.c.	-	Direct current
DCL	-	Direct coal liquefaction
DF	-	Douglas fir
DOE	-	Department of energy
Doe	-	Design of experiments

EFB	-	Empty fruit bunch
e.g.	-	For example
EI	-	Electron ionization
etc.	-	Etcetera
FFB	-	Fresh fruit bunch
GC-MS	-	Gas chromatography-mass spectrometer
GGS	-	Global green synergy
GHz	-	Giga Hertz
i.e.	-	that is, latin (id est)
ICL	-	Indirect coal liquefaction
IEA	-	International energy agency
KF	-	Karl-Fischer
LPM	-	Liter per minute
Max.	-	Maximum
MB	-	Mukah Ballingain
MEIH	-	Malaysia energy information hub
Min.	-	Minimum
MMW	-	Multimode microwave
MPOB	-	Malaysian palm oil board
Mtoe	-	Metric tonnes of oil equivalent
MS	-	Mass spectroscopy
MSD	-	Mass selective detector
MSE	-	Mean square error
MSR	-	Mean square regression
MTIB	-	Malaysian timber industry board
MW	-	Microwave
MWA	-	Microwave absorber
na	-	not available
NEB	-	National energy balance
NG	-	Natural gas
NIST	-	National institute of standards and technology
OPF	-	Oil palm fiber
OPS	-	Oil palm shell

PAHs	-	Poly aromatic hydrocarbons
Ref.	-	Reference
psi	-	Pounds per square inch
ROM	-	Run of mine
RSM	-	Response surface methodology
RSME	-	Root mean square error
SCBMs	-	Solid carbon based materials
SIM	-	Selected ion monitoring
SSE	-	Sum of square error
SSR	-	Sum of square regression
SST	-	Sum of square total
TCD	-	Thermal conductivity detector
TGA	-	Thermogravimetric analysis
TOCs	-	Total organic carbons
TPEC	-	Total primary energy consumption
TPES	-	Total primary energy supply
VOCs	-	Volatile organic carbons
WCA	-	World coal association

LIST OF SYMBOLS

A	-	Amperes
µA	-	Micro Amperes
^{o}C	-	Degree Celsius
min.	-	Minute
s	-	Second
$^{o}C/min$	-	Degree Celsius per minute
cm	-	Centimetre
cm^3	-	Cubic centimetre
cm^3/min	-	Cubic centimetre per minute
D_p	-	Penetration depth
eV	-	Electron volt
g	-	Grams
h	-	Hours
$J/^{o}C$	-	Joules per degree Celsius
L	-	Liter
µL	-	Micro Liter
L/g	-	Liter per gram
L/min.	-	Liter per minute
µL/min.	-	Micro Liter per minute
mL/min.	-	Milliliter per minute
m	-	Meter
mm	-	Millimeter
µm	-	Micrometer
m/s	-	Meter per second

mg/L	-	Milligram per Liter
M	-	Number of center points
MPa	-	Mega Pascal
n	-	Number of factors
N	-	Number of experiments
n_A	-	Number of axial points
n_F	-	Number of factorial points
Pa	-	Pascal
Q	-	Heat of water
R	-	Regression coefficient
rpm	-	Rotation per minute
t	-	Slope of the calibration curve
tan δ	-	Tangent alpha
T_{MB}	-	Temperature of middle biomass solids
T_{UBL}	-	Temperature of upper biomass layer
T_{MBL}	-	Temperature of middle biomass layer
T_{BBL}	-	Temperature of bottom biomass layer
T_{MC}	-	Temperature of middle coal solids
T_{UCL}	-	Temperature of upper coal layer
T_{MCL}	-	Temperature of middle coal Layer
T_{BCL}	-	Temperature of bottom coal layer
T_{MM}	-	Temperature of middle mixed solids
T_{UML}	-	Temperature of upper mixed layer
T_{BML}	-	Temperature of bottom mixed layer
T_{MML}	-	Temperature of middle mixed layer
v/v	-	Volume per volume
%	-	Percent
vol%	-	Volume percent
W	-	Watts
wt%	-	Weight percent
&	-	and
α	-	Alpha
Δ	-	Delta

π	-	Greek symbol Phi
$\approx$	-	nearly equals to
$<$	-	Less than
$>$	-	Greater than
$\leq$	-	Less than and equals to
$\sum$	-	Summation
Y_i	-	Predicted response
x_i	-	Independent variables
b_o	-	Intercept coefficient
b_i	-	Coefficient for linear effects
b_{ii}	-	Coefficient for quadratic effects
b_{ij}	-	Coefficient for interaction effects
λ_o	-	Incident microwave frequency\
ε'	-	Dielectric constant or real permittivity
ε''	-	Dielectric loss factor or imaginary permittivity
ε_r'	-	Relative dielectric constant
ε_o	-	Permittivity of free space

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The conventional fossil fuel oil and Natural gas (NG) fuel resources have been playing a vital role in shaping the socioeconomic status of many societies. The conventional fuel resources are valuable supply of finite natural energy. However, their increasing supply and exploited production have shown great concern over fuel source depletion (BP, 2013). In addition, increasing contribution of fossil fuels has augmented CO_2 emission. Presently, the biggest challenge faced by the oil and gas sector is how to sustain production from currently producing fields (IEA, 2010). The concern over energy insecurity and increased global CO_2 emission from fossil fuel utilization is driving many societies to look for sustainable and clean energy supply. In addition, global warming resulted from increased dependency on fossil fuel demands mitigation measures, such as a comprehensive switch to renewable energy resources.

Socioeconomic growth is vulnerable to oil and NG resources especially for the industrialized country like Malaysia. Industrial and transportation sector of the country remain heavily dependent on oil and NG (NEB, 2012). Moreover, the energy demand is increasing in the industrial sector due to its rapid growth. Currently, the most critical challenge faced by the energy sector is how to supply continuous energy and diversification of various energy resources. Malaysia total primary energy supply (TPES) reached to 81.23 Metric tonnes of oil equivalent (Mtoe) in end-2012, an increase of 163% over end-2000 (IEA, 2014b). The total primary energy consumption (TPEC) of the country was estimated at 76.3 Mtoe in end-2012 (BP, 2013). Industrial sector remained the dominant energy consumer with highest share of 43.9% to the TPEC in end-2011 (NEB, 2011). Furthermore, the future energy demand of the country is expected to grow at the annual growth rate of 5-7.9% for the next 20 years (Ong *et al.*, 2011).

More importantly, the life expectancy of Malaysia fossil fuel reserves is alarming. Oil is the fastest depleting fuel with estimated reserves available for the next 15.4 years, whereas NG reserves will be sufficient for 19.9 years of production based on reserves to production data of end-2012 (BP, 2013). Malaysia energy insecurity is growing due to its limited and quickly depleting oil and NG reserves. Energy security is a challenging issue and its insecurity may be reduced; by decreasing dependence on oil and NG through balance utilization of indigenous energy resources and their mix, substituting oil and NG with coal and renewable sources, such as bio-fuels and waste (Jain, 2011; Ong *et al.*, 2011). For this reason, the most considerable option may be to utilize indigenous fuel resources other than oil and NG and to explore potential alternatives and sustainable fuel energy supplies.

Coal still remains the largest and cheapest source of solid fuel across the globe (WCA, 2014a; WCA, 2014b). Moreover, with the increased demand of liquid transportation fuels can renew interests in coal. Nonetheless, liquid and gas fuel products from coal can become a promising option to supplement crude oil and NG products. Malaysian coal reserves are estimated at 1938.4 million tonnes (NEB, 2011). However, due to limited production from local coal resources, the country coal demands are mostly met through imports. Moreover, coal demand has increased substantially since great amount of imported coal is used in coal power plants (CPPs).

In addition, CPPs will continue to increase with the increased electricity demand for Malaysia (Othman *et al.*, 2009). Despite increased dependence on imported coal, efforts are going on to enhance coal security from local fields by exploring potential coal fields, particularly in Sarawak and from other coal fields (Ong *et al.*, 2011). The indigenous coal resources can provide an opportunity to recover synthetic fuels, which is connected to the development and exploitation of local coal resources. Nonetheless, the imported coal can be utilized to recover liquid and gas fuel products. Besides coal, biomass and waste products have captured increasing attention as a renewable energy resource.

Biomass and waste resources have been recognized as the world renewable energy to supplement the declining fossil fuel resources. The use of biomass is particularly interesting since it can reduce CO_2 emission. Malaysia is blessed with 17.98 million hectares of natural forest and 7.61 million hectares of agriculture land (MTIB, 2012). The world 46% of palm oil comes from this land and requires huge cultivation of palm oil trees (MPOB, 2014). The agro-industrial palm oil sector generates considerable quantity of waste biomass of enormous quantity, such as empty fruit bunch (EFB), mesocrap fibre, oil palm shell (OPS) and palm oil mill effluent. For this reason, proper utilization of solid waste biomass is necessary for environmental and economic reasons. In addition, the solid palm oil waste biomass, which is considered of no economic value, is commonly leftover around the palm oil mill surrounding area to decompose naturally or burned without energy recovery. Therefore, solid palm waste biomass appears the most promising and potential renewable feedstock available for the conversion into valuable fuels and energy products.

Coal and waste biomass can be utilized through combustion, gasification and pyrolysis methods. Among these, fast pyrolysis has emerged as the most promising technology to convert materials to liquid fuels at shorter duration. In spite of the various developments and improvements in the fast pyrolysis systems, it still faces some technical challenges in improving process energy efficiency, liquid yield and its quality (Bridgwater, 2012a; Bridgwater, 2012b). Microwave (MW) assisted pyrolysis is a promising attempt to resolve these challenges because of rapid and efficient internal and volumetric heating of solid materials. During MW assisted pyrolysis, the solid biomass material is rapidly heated to moderately high temperature in the presence of suitable microwave absorber (MWA) to produce oil with significant value (Bu *et al.*, 2012; Abubakar *et al.*, 2013), which can then be used to recover fuels and chemical feedstock. Besides oil, MW assisted pyrolysis of biomass produces gas (Domínguez *et al.*, 2007c; Fernández and Menéndez, 2011) and char (Salema and Ani, 2011b) with significant application values. The non-condensable gases are high content syngas readily combustible or can be used to recover heating gas components (Huang *et al.*, 2010; Ren *et al.*, 2014). Moreover, the MW assisted pyrolysis bio-char is of high carbon content material, which can be used either in soil enrichment (Lehmann and Joseph, 2009) or as a MWA (Salema and Ani, 2011b). More importantly, MW assisted pyrolysis derived coal-char can serve as feedstock for gasification/combustion process due to its high porosity (Gasner *et al.*, 1986).

Most solid material cannot be heated to desired pyrolysis temperature due to their low MW absorption capacity. Coal is essentially transparent to MW energy due to its fairly low MW assimilation capacity. This low MW absorption ability of coal signify as the major stumbling block during MW assisted pyrolysis process, but inherent moisture and mineral contents within the coal matrix responds more readily to MW due to their high MW absorption capacity (Marland *et al.*, 2001). Moreover, the coal sample cannot attain sufficient pyrolysis temperature without MWA even in the presence of high MW power source (Liu and Xia, 2012). However, carbonaceous (Cha and Kim, 1993b), metal oxides (Monsef-Mirzai *et al.*, 1992; Monsef-Mirzai *et al.*, 1995; Wang *et al.*, 2013a) and metals (Gasner *et al.*, 1986; Basheer *et al.*, 2010) absorbers are capable of increased MW assimilation capacity, which can be added to coal to achieve quick pyrolysis conditions.

The major problems associated of using metal oxides with coal solids during MW assisted pyrolysis have been highlighted by (Monsef-Mirzai *et al.*, 1995). These includes; difficulty in monitoring and controlling the pyrolysis temperature, soot formation, separation of metal oxides from coal-char and intense plasma formation. Moreover, in the presence of metals, coal solids demonstrate extremely high temperature (Basheer *et al.*, 2010), which needs specialized reactor and temperature monitoring conditions. Recently, MW assisted pyrolysis of coal in the presence of Fe_3O_4 was observed to transform metal oxide to elemental state, which was reported to limit their industrial applications (Wang *et al.*, 2013a). Besides metal oxides and metal absorbers, only few studies used carbon-based MW absorber with coal under MW pyrolysis environment (Liu and Xia, 2012; Song *et al.*, 2012).

Waste biomass shows some improved MW absorption capacity due to the presence of high moisture and inherent inorganic substances. However, in the absence of MWA, product yield from waste biomass require considerable MW power and time to reach pyrolysis conditions (Krieger-Brockett, 1994; Zhao *et al.*, 2011), and heating rate achieved at the expense of high MW power is low. Therefore, suitable MWA is required with waste biomass, which can improve process heating rate and conversion by applying low MW power source. Certain carbon based materials are capable of converting good amount of MW energy to thermal energy, which can then be transmitted to the supported waste biomass solids to improve pyrolysis conditions (Menéndez *et al.*, 2010).

Single and multimode microwave (MMW) cavities have been applied to treat various solids. Single mode MW cavities offer well-defined electric field to treat smaller volume of material, whereas MMW cavity permits electric field to encompass much larger volume of material. The geometry in MMW cavities is such that constructive and destructive interference of the MW by the cavity walls and conductor elements of the sample offers no well-defined electric field, which can be helpful to treat relatively large volume of material. Although, single mode cavities allow higher power densities, but MMW provide much greater flexibility in operation and are usually applied for the initial evaluation of the suitability of MW treatment process for particular application (Fernández *et al.*, 2011).

MMW cavities have been applied to pyrolize coal and waste biomass materials. The ON-OFF working mode in MMW cavities, variations in thermochemical properties of heterogeneous solids and non-uniform mixing method can initiate hotspot phenomena. Hotspots are likely to occur more frequently due to the strong MW interaction with the MWA material. The differential heating nature in these heterogeneous solids creates non-uniform process heating and deteriorates reaction mechanism. Several techniques has been suggested to overcome hotspots in MMW by; increasing the size of MW cavity, increasing MW frequency, rotating the material and using multiple MW inputs (Thostenson and Chou, 1999).

Previously, Salema and Ani (2011b) studied fluidized MMW pyrolysis heating of OPS with bio-char (used as MWA) and observed significant difference of sample bed and surface temperature. Later, Salema and Ani (2012a) used over-head stirrer to improve pyrolysis heating performance of OPS with coconut based activated carbon (CAC) used as MWA under fluidized MW irradiation conditions. The study suggested that the agitation of heterogeneous solids can contribute to uniform process heating with better product quality. However, the use of over-head stirrer resulted in some improved heating of sample bed and surface temperature over small time scale, but it did not contribute to complete uniformity of process heating.

In continuation of efforts to achieve uniformity of process temperature, Abubakar *et al.* (2013) used variable stirrer speed during fixed bed pyrolysis of OPS with CAC solids, which achieved nearly complete uniformity of surface and bed temperature with slow stirrer speed. It was espoused that uniformity of process temperature can improve pyrolysis performance. In another study, Chen *et al.* (2008) carried out MW heating of sawdust with silicon carbide (SiC) (a MWA) and minor inorganic additives under pyrolysis condition by distributing 15 layers of sawdust over 15 layers of SiC to observe uniform process heating. However, the final bed temperature measured at the end of pyrolysis experiment by the portable thermocouple was found higher than that measured by the pyrometer. Moreover, sufficient data of real time process heating and its effects on pyrolysis behaviour is still lacking in their study.

Above all, the most promising option is to co-utilize coal and waste biomass resources. Co-utilization can reduce some coal based pollutants, such as nitrogen oxides (NOx), sulphur oxides (SOx), poly aromatic hydrocarbons (PAHs), volatile organic carbons (VOCs), and total organic carbons (TOCs) (Chao *et al.*, 2008). Co-combustion and co-pyrolysis process are utilized to recover energy products from coal and waste biomass. Co-combustion can provide efficient and inexpensive utilization of waste biomass with coal. The co-firing or co-combustion of coal and biomass mixtures have been studied extensively (Yang *et al.*, 2012; Kubacki *et al.*, 2012; Riaza *et al.*, 2012; Wu *et al.*, 2013; Haykiri-Acma *et al.*, 2013). The co-firing technology (CFT) has been successfully demonstrated at commercial scale in various boiler types (Al-Mansour and Zuwala, 2010; Basu *et al.*, 2011). However, only fewer CFT has been demonstrated for long-term test runs (Baxter, 2005). Moreover, the major technical challenges associated with CFT are; fuel storage and preparation, transportation, ash disposal, fuel conversion, corrosion, and fly-ash utilization (Baxter, 2011).

The second option to co-combustion is co-pyrolize coal with waste biomass material. During co-pyrolysis, waste biomass can serve as inexpensive source of alkali metal catalyst and can supply hydrogen from waste biomass to coal, which may upgrade the coal fuel products (Moghtaderi *et al.*, 2004; Zhang *et al.*, 2007). Based on these facts, co-pyrolysis of coal with waste biomass may not only prospect to improve energy products but also a step towards sustainable and clean coal utilization. Early co-pyrolysis studies on coal and waste biomass mostly concentrated on the mechanism of gas production and resulted in lack of synergy (i.e. chemical interaction between coal and waste biomass) in the gas phase species (Nikkhah *et al.*, 1993; Collot *et al.*, 1999; Pan *et al.*, 1996).

The synergetic effects of coal-biomass blends on the yield of major pyrolysis products, particularly on volatile matter have been studied by Meesri and Moghtaderi (2002), Blesa *et al.* (2003), Vuthaluru (2004a), Vuthaluru (2004b) and Jones *et al.* (2005), and recently by Weiland *et al.* (2012), Bing *et al.* (2012) and Fei *et al.* (2012). These studies also reported lack of synergy. However, the synergy in terms of sulfur gas loss improved some coal de-sulfurization features (Blesa *et al.*, 2003; Cordero *et al.*, 2004) and sulfur fixing potential in coal-char (Haykiri-Acma and Yaman, 2007). Moreover, most co-pyrolysis studies were demonstrated using Thermogravimetric analysis (TGA), some in fluidized bed and entrained bed reactors. Conventional co-pyrolysis fixed bed TGA reactors provide good contact of biomass and coal particles at low heating rate, whereas high heating rate can be achieved in fluidized and entrained bed reactors, but dispersion of particles can result in very slight synergies. Therefore, conventional co-pyrolysis fixed bed TGA, fluidized, and entrained bed reactors are not suitable to investigate synergy under high and low heating rate conditions (Yuan *et al.*, 2012).

The lack of synergy may be attributed to conventional co-pyrolysis heating systems. The coal particles require sufficiently high temperature for de-volatilization compared to waste biomass. More importantly, the volatiles from waste biomass will be released at lower temperature and subsequently leaves the conventional reaction system, while the volatiles from coal will be released at higher temperature. Therefore, conventional co-pyrolysis heating systems are unable to achieve strong synergy in the vapor phase species.

1.2 Problem Statement

Most MW assisted pyrolysis techniques developed previously mainly focused towards intimately mixing of MWA with pyrolysis solids (Salema and Ani, 2012a;

Abubakar *et al.*, 2013; Bu *et al.*, 2013; Bu *et al.*, 2014) with much less attention towards uniform distribution method. The non-uniform distribution of MWA with pyrolysis solids or intimately mixing of heterogeneous solids under MW irradiation conditions initiate hotspots phenomena (a high temperature region formed where the MW energy is more frequently absorbed by the MWA than the surrounded pyrolysis material). These hotspots create non-uniform process heating and deteriorate pyrolysis reaction mechanism. For this reason, the distribution of MWA with pyrolysis material needs proper attention to design an energy efficient system to minimize and control hotspots. For this study, it was hypothesized that the uniform distribution of MWA over pyrolysis material by layer-to-layer arrangement method can control the hotspots to MWA layer, which can contribute to uniform process heating. In addition, it was suggested that MW energy absorption by the uniformly distributed MWA layer and simultaneous heat transfer to supported pyrolysis layer can improve pyrolysis heating performance.

The motivation to use waste biomass with coal is growing. Researchers are now focused on how to exploit biomass high volatile matter and alkali metal contents to upgrade fuel components when co-utilized with coal. The conventional co-utilization of coal and waste biomass resources through co-pyrolysis routes offers some benefits over SO_x (Blesa *et al.*, 2003; Cordero *et al.*, 2004) and NO_x emission (Yuan *et al.*, 2011), but most studies observed additive fuel behaviour in blends (Masnadi *et al.*, 2014; Guan *et al.*, 2015) and were unable to achieve strong synergy in vapor phase species. It was established that the MW assisted pyrolysis of coal and biomass with carbon based MWA in isolation offers number of advantages over conventional pyrolysis system (Domínguez *et al.*, 2007c; Menéndez *et al.*, 2010; Song *et al.*, 2012; Yin, 2012).

Despite improved heating nature and fuel recovery from MW assisted pyrolysis of isolated fuels with carbons, most co-pyrolysis studies remained limited to conventional systems only. For this study, it was therefore proposed that MW assisted co-pyrolysis of coal and biomass over carbon surfaces is considered effective for vapor phase synergies. Nonetheless, to date, co-utilization of coal and biomass resources has not been investigated for vapor-phase synergy in MW pyrolysis reactor.

1.3 Objectives of the Study

The primary aim of this study was to control hotspots by uniformly distributed CAC solids over OPS and coal solids in isolation to investigate its effects on MW heating performance, and to study the MW assisted co-pyrolysis of coal and OPS in segregation and blend with uniformly distributed CAC solids to observe vapor-phase synergy.

The specific objectives of this study are to:

i. Investigate the effects of uniformly distributed CAC to control hotspots over OPS solids on pyrolysis system performance at various levels of CAC loading, MW power and N_2 flow rate.

ii. Examine the effects of uniformly distributed CAC to control hotspots over coal solids on pyrolysis system performance at various levels of CAC loading, MW power and N_2 flow rate.

iii. Study the MW assisted co-pyrolysis of coal and OPS in segregation and blend with uniformly distributed CAC solids at various levels of CAC loading to observe vapor phase synergy.

iv. Optimize oil, char and gas production from MW assisted co-pyrolysis of coal and OPS, and to study the significance and interaction effects among the process factors on product yield.

1.4 Scopes of the Study

The scopes of this study are based on the objectives and are as follow:

i. The OPS waste biomass and Mukah Ballingain (MB) coal were used as the primary feedstocks in pyrolysis and co-pyrolysis MW heating environment.
ii. MMW heating cavity was utilized to treat coal and OPS solids.
iii. The CAC solids were used as MWA with OPS and coal.
iv. The CAC solids were uniformly distributed over OPS and coal solids.
v. MW assisted pyrolysis of OPS and coal without and with uniformly distributed CAC solids in isolation were carried out at various levels of CAC loading (wt%), MW power (W) and N_2 flow rate (LPM).
vi. MW assisted co-pyrolysis of coal and OPS were carried out in segregation (upper-bed-OPS/bottom-bed-coal and upper-bed-coal/bottom-coal-OPS) and blend (1:1) with uniformly distributed CAC solids.
vii. MW assisted co-pyrolysis oil, char and gas produced with blend fuel in the presence of uniformly distributed CAC solids were optimized with three process factors; CAC loading (wt%), MW power (W) and N_2 flow rate (LPM).

1.5 Significance of the Study

MW energy has found widespread application in the treatment of various heterogeneous solids, particularly involving carbon solids: ranging from metallurgical and mineral processing (Marland *et al.*, 2000; Uslu and Atalay, 2004; Yoshikawa *et al.*, 2007), biomass valorization (Salema and Ani, 2011b; Salema and Ani, 2012a), bio-solids handling (Menendez *et al.*, 2002; Beneroso *et al.*, 2014), soil decontamination (Li *et al.*, 2008; Xu and Lee, 2008), to the carbon catalyzed gas-phase reactions, such as NOx and SOx reduction (Cha and Kim, 2001) and CH_4 decomposition to H_2 production (Domínguez *et al.*, 2007b; Fidalgo *et al.*, 2008b). More importantly, carbon solids have been found effective in in-situ upgrading of pyrolysis vapors to value-added chemicals under MW irradiation conditions (Bu *et al.*, 2011; Abubakar *et al.*, 2013). The reason behind this diverse application is carbons are capable of converting good amount of MW energy to thermal energy, which can then be transmitted to the supported materials.

The use of carbons in MW heating process offers number of advantages, but with some serious technical challenges, such as non-uniform process heating, difficulty in measuring process temperature and hotspots formation (Luque *et al.*, 2012). These hotspots are thermally unstable regions created as a result of nonlinear dependence of MW and the variations in thermo-electromagnetic properties of the materials being heated. In MW treatment of heterogeneous solids, the rate at which the MW energy is absorbed by the MWA solids is much higher than the supported material. As a result, a region of very high temperature can form called hotspots. These localized hotspots can result in non-uniform process heating and deteriorate reaction mechanism. For this reason, the distribution of carbon absorber with supported material needs proper attention to design an energy efficient system to minimize and control hotspots.

The results of this study suggests that pyrolysis and co-pyrolysis heating profiles of pyrolysis solids with uniformly distributed carbon solids under MW irradiation environment did not significantly affected MW penetration depth. In addition, this study measured true and real time temperature profiles of pyrolysis solids by placing the thermocouples into the pyrolysis solids, which otherwise was difficult in intimately mix method since the thermocouple tip may directly come in contact with carbon solids or hotspots region. These high temperature regions were observed to meltdown the thermocouple when carbon absorber was intimately mixed with OPS solids and subjected to MW irradiation conditions during preliminary studies. Nonetheless, the layer-to-layer arrangement provides a unique method of limiting the hotspots to the carbon layers only.

More interesting, the OPS solids with carbon surfaces achieved nearly complete uniformity of process heating under controlled conditions of CAC loading, MW power and N_2 flow rate. These uniform heating conditions were observed to improve bio-oil yield. Moreover, uniform process heating in the presence of carbon surfaces improved phenol selectivity with highest detected 71.77% GC-MS area. More importantly, single surface carbon solids at 35 wt% carbon loading over OPS-nuts with 300 W and 4 LPM of N_2 flow rate generated quick pyrolysis conditions. This finding suggests that the heat generated from carbon surface and carried with N_2 gas can improve pyrolysis heating conditions at fairly low MW power, which can save process energy. The coal-tar obtained were complex mixture of several groups of chemicals. However, higher aromatics (napthalenes, benzenes and xylene) and saturated aliphatics (alkanes and alkenes) hydrocarbons in coal-tar needs downstream refining for fuel recovery.

More importantly, the total bed height of segregated fuels in MW assisted co-pyrolysis of coal and OPS solids was measured 10.4 cm, 12.2 cm and 14 cm with 35 wt%, 55 wt% and 75 wt% carbon loading, respectively. Despite the increased bed height, the co-pyrolysis heating profiles of pyrolysis solids showed improved process heating rate and final pyrolysis temperature. This finding suggests that MW pyrolysis system can be operated with higher solid loading. The co-pyrolysis oil obtained with segregated and blended fuel methods were dominated with polars (phenol, phenolics and guaiacols) compounds. However, upper-bed-coal/bottom-bed-OPS segregated contact method produced much higher polar compounds compared to upper-bed-OPS/bottom-bed-coal and blended fuel methods. More importantly, the formation of aromatics (naphthalenes, benzenes and xylene) and saturated aliphatics (alkanes and alkenes) detected were observed lowest with upper-bed-coal/bottom-bed-OPS arrangement. This improved polar formation with limited aromatics and saturated aliphatics suggests that the presence of carbon surfaces and bio-char in the bottom-bed catalyzed and enhanced vapor-phase synergy.

1.6 Thesis Structure

The research frame work of this thesis is depicted in Figure 1.1. This thesis is organized into eight (8) chapters and the contents of the chapters are:

i. Chapter 1 contains the overview of the research background, problem statement, objectives, scopes and significance of this study.

ii. Chapter 2 starts with introduction of coal and palm oil biomass resources of Malaysia and its potential. The physico-chemical properties of coal and palm oil waste biomass, and various energy recovery methods are reviewed. The improved features of MW pyrolysis heating over convectional pyrolysis system are discussed. The MW assisted pyrolysis of coal with carbonaceous and metal oxide absorbers, and metals, and MW assisted pyrolysis of waste biomass without absorber and with carbonaceous absorbers, inorganic and metal oxide additives are reviewed. The co-utilization of coal and biomass through conventional co-gasification and co-pyrolysis routes are reviewed. Finally, MW assisted pyrolysis factor considerations and concerns are summarized by analyzing their association on process temperature, heating rate, product yield and composition.

iii. Chapter 3 explains the methodology of this study which includes, sample collection and preparation, pyrolysis and co-pyrolysis factor selection, MW power calibration, experimental setup, pyrolysis and co-pyrolysis sample preparation and methods, and experimental design to optimize products and to model the co-pyrolysis process factors. The details of the product analysis and methods done are given at the end of the chapter.

iv. Chapter 4 thoroughly discusses the results of fixed bed pyrolysis behaviour of OPS without and with uniformly distributed CAC solids under MW irradiation conditions. The effects of process variables on biomass heating profile, pyrolysis product yield and chemical composition are reported.

v. Chapter 5 highlights the detailed discussion on fixed bed pyrolysis behaviour of coal without and with uniformly distributed CAC under MW irradiation conditions. The effects of process variables on coal heating profile, pyrolysis product yield and chemical composition are reported.

vi. Chapter 6 presents a comprehensive data analysis of fixed bed co-pyrolysis behaviour of coal and OPS solids in segregation and blend methods with uniformly distributed CAC under MW irradiation conditions. A detailed discussion on MW assisted co-pyrolysis heating profile, product yield and chemical composition of the oil obtained is presented to observe vapor-phase synergy.

vii. Chapter 7 details the results and discussion on optimization of products from MW assisted co-pyrolysis of OPS and coal with blend method. The response surface methodology was used to establish model yields. The chemical composition of co-pyrolysis oil obtained under MW operating conditions was analyzed using GC-MS.

viii. Chapter 8 revisits the hypothesis and concludes with the important findings of this research work. The benefits of this study are presented. The recommendations for future work are suggested lastly.

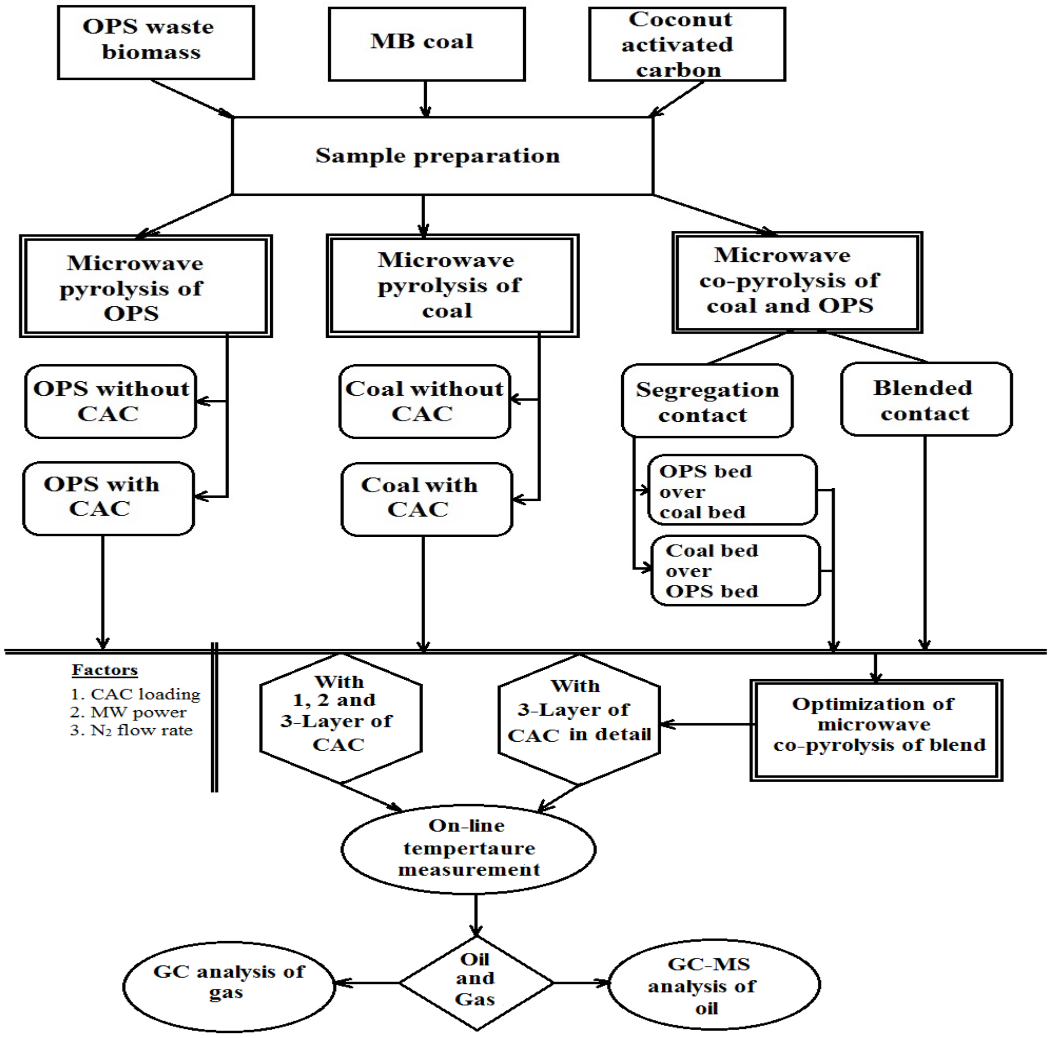

Figure 1.1: Research framework of MW assisted pyrolysis and co-pyrolysis of coal and OPS

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