

PARAMETRIC AND KINETIC STUDIES OF MICROWAVE –ASSISTED
PYROLIGNEOUS ACID FROM OIL PALM FIBER

FATIMATUL ZAHARAH ABAS

UNIVERSITI TEKNOLOGI MALAYSIA

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FATIMATUL ZAHARAH ABAS

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Faculty of Engineering
Universiti Teknologi Malaysia

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ABSTRACT

Malaysia is acknowledged as the world's second largest producer and exporter of palm oil. This in turn has generated large amount of oil palm biomass which is normally left at the plantation to putrefy, burnt or turned into various low-value products. Hence, an alternative approach which is more robust, easier and environmental friendly to process this biomass is required. Microwave-assisted pyrolysis allows the conversion of biomass to various pyrolytic products for example pyrolygneous acid (PA), biofuel, biogas, and biochar. However, to date, there are still limited reports available on the application of microwave-assisted pyrolysis for the production of PA in optimized condition, which is the novelty of this study. The objective of this study was to optimize the operating conditions during the microwave-assisted pyrolysis of oil palm fiber (OPF) in order to obtain the highest total phenolic content (TPC) as well as the highest liquid yield of microwave-assisted pyrolygneous acid (MWPA). The optimization process was performed using central composite design technique via response surface methodology which focused on the following parameters; final temperature (400-600 °C), holding time (15-30 min) and activated carbon (AC) loading (50-100 g). MWPA was characterized for its chemical and antimicrobial properties. The thermal degradation profiles of OPF were evaluated using the Kissinger-Akahira Sunose (KAS), Ozawa-Flynn wall (OFW) and Coats-Redfern method (CRM) kinetic models based on the thermogravimetric analysis data. The optimum condition to produce the highest TPC and liquid yield of MWPA were as follows; 540 °C, holding time of 23 min and AC loading of 86.74 g. The following profiles were determined for concentrated MWPA extracted with ethyl acetate (C-MWPA); antioxidant properties of $18.52 \pm 0.2\%$ with IC_{50} of $285.66 \mu\text{g/ml}$ (2,2-diphenyl-1-picrylhydrazyl radical scavenging), $0.836 \pm 0.003 \text{ mM}$ (ferric reducing antioxidant power), $12 \pm 0.07\%$ (metal chelating activity), $414.21 \pm 4.74 \mu\text{mol ascorbic acid/g}$ (phosphomolybdenum), $85.42 \pm 0.33\%$ (hydrogen peroxide assay), and growth-inhibition of $28.67 \pm 0.88 \text{ mm}$ towards *Escherichia coli* American Type Culture Collection 25922 with minimum inhibition concentration value of $0.651 \pm 0.13 \text{ mg/ml}$. The kinetics study evaluated for OPF found that the mean value of activation energy (E_a) were 97.01 kJ/mol (KAS) and 101.52 kJ/mol (OFW). The values of E_a calculated for C-MWPA were 28.59 kJ/mol and 33.87 kJ/mol for KAS and OFW, respectively. The validation of kinetic reaction model by using CRM model demonstrated that, OPF thermal degradation was well represented by the three dimension diffusion reaction model with mean value E_a of 84.71 kJ/mol while C-MWPA was well fitted with second order reaction type (E_a of 39.83 kJ/mol). To conclude, this study has successfully demonstrated the application of microwave-assisted pyrolysis to convert oil palm biomass into various useful pyrolytic products. This is considered as a significant finding as this technique can offer a substantial reduction in processing time, able to process large volumes at a given time, easy to operate as well as offering various valuable pyrolytic products.

ABSTRAK

Malaysia diiktiraf sebagai pengeluar dan pengeksport minyak kelapa sawit kedua terbesar dunia. Keadaan ini sebaliknya telah menghasilkan banyak sisa biojisim kelapa sawit yang selalunya dibiarkan di ladang untuk mereput, dibakar atau diubah menjadi pelbagai produk bernilai rendah. Oleh itu, ianya memerlukan pendekatan alternatif yang lebih mantap, mudah dan mesra alam sekitar. Pirolisis berbantuan gelombang mikro membolehkan penukaran biojisim kepada pelbagai produk pirolisis seperti asid piroligneus (PA), bio-bahanapi, bio-gas, dan bio-arang. Walaubagaimanapun, sehingga hari ini, kajian penggunaan pirolisis pemanasan berbantuan gelombang mikro bagi penghasilan PA dalam keadaan optimum masih terhad, yang merupakan pembaharuan dalam kajian ini. Objektif kajian ini adalah untuk mengoptimalkan keadaan operasi semasa pirolisis berbantuan gelombang mikro serat kelapa sawit (OPF) bagi tujuan memperoleh jumlah kandungan fenolik (TPC) dan juga hasil cecair asid piroligneus berbantuan gelombang mikro (MWPA) yang tertinggi. Proses pengoptimuman telah dilakukan menggunakan teknik rekabentuk komposit berpusat melalui kaedah tindakbalas permukaan dengan menumpukan kepada beberapa parameter seperti berikut; suhu akhir (400-600 °C), masa pegang (15-30 min) dan muatan karbon teraktif (AC) (50-100 g). Sifat-sifat kimia dan anti-bakteria MWPA telah dianalisa. Profil degradasi termal OPF telah dianalisa menggunakan model kinetik *Kissinger-Akahira Sunose* (KAS), *Ozawa-Flynn wall* (OFW) dan *Coats-Redfern method* (CRM) berdasarkan kepada data analisa termogravimetri. Keadaan optimum untuk menghasilkan TPC dan hasil cecair MWPA yang tertinggi adalah seperti berikut; 540 °C, 23 min masa pegang dan 86.74 g muatan AC. Berikut adalah profil yang telah dianalisa untuk kepekatan MWPA yang diekstrak dengan etil asetat (C-MWPA); sifat-sifat antioksidan iaitu $18.52 \pm 0.2\%$ dengan IC_{50} sebanyak $285.66 \mu\text{g/ml}$ (*2,2-diphenyl-1-picrylhydrazyl radical scavenging*), $0.836 \pm 0.003 \text{ mM}$ (*ferric reducing antioxidant power*), $12 \pm 0.07\%$ (*metal chelating activity*), $414.21 \pm 4.74 \mu\text{mol}$ asid askorbik/g (fosfomolibdenum), $85.42 \pm 0.33\%$ (*hydrogen peroxide assay*), dan rencatan pertumbuhan MWPA terhadap *Escherichia coli American Type Culture Collection* 25922 adalah $28.67 \pm 0.88 \text{ mm}$ dengan nilai kepekatan rencatan minimum sebanyak $0.651 \pm 0.13 \text{ mg/ml}$. Kajian kinetik yang dikira untuk OPF mendapati bahawa nilai purata tenaga pengaktifan (E_a) adalah 97.01 kJ/mol (KAS) dan 101.52 kJ/mol (OFW). Nilai E_a bagi C-MWPA ialah 28.59 kJ/mol dan 33.87 kJ/mol bagi KAS dan OFW. Pengesahan dari analisa tindak balas kinetik menggunakan kaedah CRM menunjukkan degradasi termal OPF mempunyai model tindak balas penyebaran tiga dimensi dengan nilai purata E_a sebanyak 84.71 kJ/mol , manakala C-MWPA menunjukkan jenis tindak balas kedua (E_a sebanyak 39.83 kJ/mol). Kesimpulannya, kajian ini telah berjaya menunjukkan penggunaan pemanasan berbantuan gelombang mikro dapat menukarkan biojisim kelapa sawit kepada pelbagai produk pirolisis yang berguna. Penemuan hasil kajian ini penting kerana teknik ini menyebabkan pengurangan yang besar dalam masa pemprosesan, berkemampuan memproses dalam jumlah besar pada waktu tertentu, operasi yang mudah serta dapat menghasilkan pelbagai produk pirolisis berharga.

TABLE OF CONTENTS

CHAPTER	TITLE	PAGE
	DECLARATION	ii
	DEDICATION	iii
	ACKNOWLEDGEMENT	iv
	ABSTRACT	v
	ABSTRAK	vi
	TABLE OF CONTENTS	vii
	LIST OF TABLES	xiii
	LIST OF FIGURES	xvi
	LIST OF ABBREVIATIONS	xx
	LIST OF SYMBOLS	
	xxiv	
	LIST OF APPENDICES	xxvii
1	INTRODUCTION	1
	1.1 Research Background	1
	1.2 Problem Statement	5
	1.3 Research Objectives	6
	1.4 Scope of Study	7
	1.5 Significance of Study	8
2	LITERATURE REVIEW	9
	2.1 Oil Palm Plantation	9
	2.2 Oil Palm Biomass and Management Strategies	11
	2.2.1 Types of Oil Palm Biomass	11

2.2.2	Biomass Management Strategies	13
2.3	Lignocellulosic Biomass	14
2.3.1	Lignin	16
2.3.2	Cellulose	18
2.3.3	Hemicellulose	19
2.4	Pyrolysis	21
2.4.1	Pyrolysis Process	21
2.4.2	Factors Influencing Pyrolysis Process	24
	2.4.2.1 Final Heating Temperature	25
	2.4.2.2 Amount of AC loading	25
	2.4.2.3 Holding Time	26
	2.4.2.4 Particle Size of Material	27
	2.4.2.5 Heating Rate	27
2.5	Pyroligneous Acid	28
2.5.1	Production of Pyroligneous Acid	28
2.5.2	Liquid-liquid Extraction of Pyroligneous Acid	29
2.5.3	Composition of Pyroligneous Acid	30
2.5.4	GC-MS Analysis of Pyroligneous Acid	32
2.5.5	Industrial Application of Pyroligneous Acid	34
2.6	Oil Palm Fiber	35
2.7	Microwave Heating	37
2.7.1	Fundamental of Microwave Heating	37
2.7.2	Microwave-Assisted Pyrolysis Heating	38
2.7.3	Pyrolytic Product of Microwave-assisted Pyrolysis Heating	40
2.8	Antioxidant Effect of Pyroligneous Acid	45
2.8.1	Antioxidant Assay	46
	2.8.1.1 DPPH Free Radical Scavenging Activity	46
	2.8.1.2 Metal Chelating Activity	48
	2.8.1.3 Ferric Reducing Antioxidant Power	49
	2.8.1.4 Phosphomolybdenum Assay	49
	2.8.1.5 Hydrogen Peroxide Assay	50
2.9	Antimicrobial Properties of Pyroligneous Acid	50
2.9.1	Efficiency of Pyroligneous Acid as Antimicrobial Agent	50

2.9.2	Antimicrobial Activity Assay	52
2.9.2.1	Disk Diffusion Method	52
2.9.2.2	Minimum Inhibitory Concentration	53
2.10	Response Surface Methodology as Experimental Design	53
2.10.1	Response Surface Methodology	54
2.10.2	Central Composite Design	56
2.11	Kinetic Study and Thermodynamic Analysis	57
2.11.1	Evaluation of Kinetic Study Analysis	62
2.11.2	Ozawa-Flynn-Wall	64
2.11.3	Coats-Redfern Method	65
2.11.4	Kissinger-Akahira-Sunose	66
2.11.5	Thermodynamic Study	67
2.12	Summary of the Present Study	69
3	METHODOLOGY	70
3.1	Introduction	70
3.2	Sampling of Oil Palm Fiber (OPF)	72
3.2.1	Acid-Insoluble and Soluble Lignin Analysis	73
3.2.2	Carbohydrate Analysis in Biomass	74
3.3	Characterization of As-Received Oil Palm Fiber	76
3.3.1	Proximate Analysis	76
3.3.2	Ultimate Analysis	78
3.3.3	OPF Thermogravimetric Analysis	78
3.4	Production of Microwave-Assisted Pyroligneous Acid from Oil Palm Fiber	79
3.4.1	Experimental Design of Microwave-Assisted Pyroligneous Acid	79
3.4.2	Production of Microwave-Assisted Pyroligneous Acid	81
3.4.3	Optimization Study of Microwave-Assisted Pyroligneous Acid	81
3.5	Extraction of Microwave-Assisted Pyroligneous Acid	84
3.6	Evaluation of Total Phenolic Content	85
3.7	Characterization of Microwave-Assisted Pyroligneous Acid	86

3.7.1	Karl-Fischer Titration	86
3.7.2	Gas Chromatograph-Mass Spectroscopy (GC-MS) Analysis	87
3.8	Antioxidant Assay of Microwave-Assisted Pyroligneous Acid	88
3.8.1	DPPH Free Radical Scavenging Activity	88
3.8.2	Ferric Reducing Antioxidant Power	89
3.8.3	Metal Chelating Activity	89
3.8.4	Phosphomolybdenum Assay	90
3.8.5	Hydrogen Peroxide Scavenging Assay	91
3.9	Antimicrobial Activity Assay of Microwave-Assisted Pyroligneous Acid	91
3.9.1	Microorganism Strain and Culture	91
3.9.2	Disk Diffusion Method of Antimicrobial Assay	92
3.9.3	Relative Percentage Inhibition	94
3.9.4	Minimum Inhibitory Concentration (MIC)	94
3.9.5	Minimum Bactericidal Concentration (MBC)	95
4	RESULT AND DISCUSSION	96
4.1	Introduction	96
4.2	Characterization of Oil Palm Fiber	97
4.2.1	Lignocellulosic Content Analysis of Oil Palm Fiber	97
4.2.2	Elemental Analysis	99
4.2.3	TGA-DTG Analysis of OPF	101
4.3	Microwave-Assisted Pyroligneous Acid (MWPA)	104
4.3.1	Temperature Profile for the Production of MWPA	104
4.3.2	Total Phenolic Content and Product Distribution Yield of MWPA	107
4.3.2.1	Effect of Final Temperature	110
4.3.2.2	Effect of AC Loading	112
4.3.2.3	Effect of Holding Time	113
4.4	Optimization Process on TPC and MWPA Yield	114
4.4.1	Regression Model Analysis of TPC Concentration and PA Yield	116

4.4.1.1	Analysis of Variance on Total Phenolic Content	117
4.4.1.2	Analysis of Variance on Yield of Pyroligneous Acid	120
4.4.2	Interaction Variable by 3-D Graphical Plot	123
4.4.2.1	3-D Graphical Plot on TPC Concentration	124
4.4.2.2	3-D Graphical Plot on PA Yield	126
4.4.3	Validation of Optimize Parameter for TPC and PA Yield	128
4.5	Characterization of Optimized Microwave-assisted Pyroligneous Acid (MWPA)	131
4.5.1	GC-MS Analysis of CPAEA from MWPA (C-MWPA)	133
4.5.2	GC-MS Analysis of CPAEA from MPOB (C-MPOB)	137
4.6	Total Phenolic Content	140
4.7	Antioxidant Activity of CPAEA	141
4.7.1	DPPH Free Radical Scavenging Activity	141
4.7.2	Ferric Reducing Antioxidant Power (FRAP)	144
4.7.3	Metal Chelating Activity	145
4.7.4	Phosphomolybdenum Activity	147
4.7.5	Hydrogen Peroxide Activity	149
4.8	Antimicrobial Activity of CPAEA	151
4.8.1	Propagation of Bacteria Strain	151
4.8.2	Antibacterial Activity of CPAEA	152
4.8.3	Minimum Inhibitory Concentration	158
4.8.4	Minimum Bactericidal Concentration	161
4.9	Thermokinetic Analysis of OPF Biomass and MWPA	164
4.9.1	Thermogravimetric Analysis of OPF Biomass	164
4.9.2	Kinetic Model Analysis of OPF Biomass	168
4.9.3	Thermogravimetric Analysis of C-MWPA	177
4.9.4	Kinetic Model Analysis of C-MWPA	179
4.9.5	Validation of Kinetic Analysis for OPF Biomass and C-MWPA	187
4.9.5.1	Model Validation for OPF Biomass	187
4.9.5.2	Model Validation for C-MWPA	191

4.9.6	Kinetic Correlation Effect	193
4.9.7	Thermodynamic Study Analysis	196
4.9.7.1	Thermodynamic Properties of OPF Biomass	196
4.9.7.2	Thermodynamic Properties of C-MWPA	200
5	CONCLUSION AND RECOMMENDATION	205
5.1	Conclusion	205
5.2	Recommendation	207
	REFERENCES	208
	Publications	245
	Appendices A-K	246-265

LIST OF TABLES

TABLE	TITLE	PAGE
1.1	Characteristic comparison between gasification, combustion and pyrolysis	3
2.1	General composition of lignocellulosic biomass	15
2.2	Chemical compositions produced from pyrolysis of lignocellulosic biomass	20
2.3	Major categories of pyrolysis process	22
2.4	Various compounds present in PA	31
2.5	GC-MS analysis of PA from different feedstock	33
2.6	Recent applications of PA in industry	35
2.7	Elemental and lignocellulosic analysis of raw OPF	36
2.8	Comparison between microwave-assisted pyrolysis heating and conventional pyrolysis heating	40
2.9	Pyrolytic product obtained from microwave-assisted pyrolysis of various types of lignocellulosic biomass	41
2.10	Differences between three most common design used in RSM	55
2.11	Kinetic analysis of biomass pyrolysis by using TGA	58
2.12	Kinetic reaction models in the differential form ($f(\alpha)$) and integral form ($g(\alpha)$) that mostly applied in the kinetic study of heterogeneous solid state system	61
2.13	Comparison between three models approach for the kinetic study analysis	62
3.1	Experiment design summary for the optimization of MWPA with coded value	82
3.2	Experiment matrix of 2^3 central composite design (CCD) with coded value for the optimization of MWPA	83

4.1	Total lignin and ash content of OPF for five different oil palm mills around Johor	98
4.2	Proximate analysis profile for as-received OPF	100
4.3	Ultimate analysis profile for as-received OPF	101
4.4	Design summary for optimized production of MWPA for TPC concentration and PA yield	114
4.5	Optimization of MWPA from OPF	115
4.6	Model summary statistic on the coefficient determination	116
4.7	ANOVA summary for the TPC Concentration	118
4.8	ANOVA summary for PA yield	121
4.9	Validation of optimization constraint and solution suggested by the CCD for factor variables and responses	129
4.10	Validation test result for TPC and yield of PA at optimum condition	130
4.11	Karl-fisher titration for water content of microwave-assisted pyroligneous acid	132
4.12	Chemical constituent of C-MWPA via GC-MS analysis	134
4.13	Chemical constituent of C-MPOB via GC-MS analysis	138
4.14	Radical scavenging activity and IC ₅₀ value for all extracts	143
4.15	Inhibition zone of both CPAEA towards bacterial strains after 24 hours incubation	152
4.16	Diameter of inhibition zone by disk diffusion method for both CPAEA	155
4.17	MIC observation toward 1.5:1 of C-MWPA and 1.5:1 of C-MPOB	159
4.18	MIC value of various types of CPAEA against different bacterial strains, n=3	160
4.19	The MBC observation between 1.5:1 of C-MWPA and 1.5:1 of C-MPOB against all strains after 24 hr incubation at 37 °C	162
4.20	MBC values of various types of CPAEA against different bacteria strains, n=3	163
4.21	Temperature values which corresponding to the degree of conversion of OPF biomass at different heating rates and data related to construct KAS and OFW models	170

4.22	The fitted equation, activation energy (E_a) and correlation coefficient (R^2) for KAS and OFW models at different degree of conversion for all heating rates	172
4.23	Range of E_a values for different kind of biomass using different types of kinetic model analysis	174
4.24	The pre-exponential factor, (A) of OPF biomass for KAS and OFW models at different heating rates along the progressing conversion	175
4.25	Temperature values that corresponding to the degree of conversion of C-MWPA for all heating rates and data related to construct KAS and OFW models	180
4.26	The activation energy (E_a), fitted equation and correlation coefficient (R^2) of C-MWPA for KAS and OFW models at different conversion for all heating rates	183
4.27	The pre-exponential factor, (A) of C-MWPA for KAS and OFW models at different heating rates along the progressing conversion	186
4.28	Value of E_a calculated for different types of mechanism using CRM method for degradation of OPF biomass at different heating rates	188
4.29	Value of E_a for different types of mechanism using the CRM method for C-MWPA degradation at different heating rates	191
4.30	The thermodynamics properties during OPF biomass degradation at different degree of conversion under different heating rates	197
4.31	Data distribution of thermodynamics properties of optimized C-MWPA at different conversion and heating rates	202

LIST OF FIGURES

FIGURE	TITLE	PAGE
1.1	The main process of biomass conversion	2
2.1	Oil palm plantation area in Malaysia from 1980 to 2017	10
2.2	The major resources of oil palm biomass	11
2.3	Generation of oil palm biomass from oil palm processing at the oil palm mill	12
2.4	Cellulose strands surrounded by hemicellulose and lignin	15
2.5	Three major phenylpropanoid units of lignin; (a) trans- <i>p</i> -coumaryl alcohol, (b) trans-coniferyl alcohol and (c) trans-sinapyl alcohol	17
2.6	Chemical and physical process in biomass during pyrolysis	23
2.7	Cross sectional image of oil palm fruit showing OPF	36
2.8	Reduction of DPPH molecules	47
3.1	Research methodology flowchart for microwave-assisted pyrolygneous acid (MWPA)	71
3.2	As-received of fresh OPF in shredded form	72
3.3	Pre-processing of OPF samples collected from different oil palm mills around Johor	73
3.4	Schematic diagram of experimental set-up for microwave pyrolysis; 1) Microwave system; 2) quartz glass reactor; 3) thermocouple type-R; 4) top flange lid; 5) bottom flange lid; 6) wire mesh; 7) flowmeter; 8) N ₂ gas; 9) condenser unit; 10) collector; 11) temperature controller; 12) picolog data logger; 13) personal computer; 14) lab jack; 15) chiller; 16) biomass	80

3.5	Extraction process of pyroligneous acid by using ethyl acetate	85
3.6	Selective agar slant preparation for strain culture; Green for SCA, Orange for HBA, Red for MA as well as Light Yellow for BPA and M.R.S.A	92
3.7	The procedure of disc diffusion method for determination of inhibition zone before incubated at 37 °C for 24 hours	93
3.8	MIC determination using 96 wells microplate before incubation (1 st well to 10 th containing NB + CPAEA + Inoculum, 11 th well containing NB + Inoculum (control) and 12 th well containing NB (blank)	95
4.1	TGA-DTG curve of as-received OPF biomass	102
4.2	Microwave-assisted pyroligneous acid (MWPA) produced from OPF	104
4.3	Temperature profiles (standard 2, 11, 13, and 19) of MWPA at different final temperature, holding time and activated carbon loading	105
4.4	Microwave-assisted product distribution yield at different condition	109
4.5	The predicted versus actual value plot for TPC concentration	120
4.6	The normal plot of residual for TPC concentration	120
4.7	The predicted versus actual value plot for PA yield	123
4.8	The normal percent probability residual plot for PA yield	123
4.9	Interaction between variables by 3D surface plot on the TPC concentration	125
4.10	Interaction between variables by 3D surface plot on the yield of PA	127
4.11	Histogram solution suggested by CCD for validation test	129
4.12	Temperature profile of MWPA at optimum condition	130
4.13	Total phenolic content (TPC) in different types of PA	140
4.14	DPPH absorbance profile for C-MPOB and C-MWPA; ascorbic acid and BHA acted as control; n=3	142
4.15	DPPH scavenging activities for C-MPOB and C-MWPA; ascorbic acid and BHA acted as control; n=3	142

4.16	Ferric reducing antioxidant power of various types of CPAEA, n = 3	144
4.17	Absorbance profile of metal chelating assay for various CPAEA, n = 3	146
4.18	Percentage of metal chelating activity for various CPAEA; n=3	146
4.19	Intensity of phosphomolybdenum (V) complex when added with various concentrations of C-MPOB and C-MWPA	148
4.20	Phoshomolybdenum reducing activity and absorbance for all C-MPOB and C-MWPA, n=3	148
4.21	H ₂ O ₂ scavenging activity by various type of CPAEA, n=3	150
4.22	Bacteria cultured on selective agar plate after 24 hours incubation at 37 °C	151
4.23	Relative percentage inhibition of various CPAEA towards all bacteria strains	158
4.24	TG (a) DTG (b) curves under non-isothermal condition of OPF biomass at different heating rates	165
4.25	Decomposition of OPF with increasing temperature at different heating rates	168
4.26	Linear regression for KAS model plot curve of OPF biomass at different degree of conversion for all heating rates (10, 20 and 30 °C/min)	171
4.27	Linear regression for OFW model plot curve of OPF biomass at different degree of conversion for all heating rates (10, 20 and 30 °C/min)	171
4.28	The activation energy, (E_a) profile of OPF biomass with the progressing in degree of conversion for KAS and OFW model	173
4.29	Linear plot of CRM model for OPF biomass at different heating rates for the determination of pre-exponential factor	175
4.30	TG-DTG curve under non-isothermal condition for optimized C-MWPA at different heating rates	177
4.31	The degree of conversion of C-MWPA with increasing temperature at different heating rates	180
4.32	Linear regression curve for KAS model of C-MWPA at different degree of conversion for all heating rates	182

4.33	Linear regression curve for OFW model of C-MWPA at different degree of conversion for all heating rates	182
4.34	The activation energy, (E_a) profile of C-MWPA with the progressing degree of conversion for KAS and OFW model	184
4.35	Linear plot of CRM model for C-MWPA at different heating rates for the determination of pre-exponential factor	186
4.36	The most probable linear plots of D2, D3 and D4 reaction models for all heating rates analyzed using CRM approach for OPF biomass thermal degradation	190
4.37	The most probable linear plots for 1 st and 2 nd order reaction models for all heating rates analyzed using CRM approach for thermal degradation of C-MWPA	192
4.38	The linear plot of KCE effect for (a) OPF biomass and (b) C-MWPA	195

LIST OF ABBREVIATIONS

AA	-	Ascorbic acid
ABTS	-	2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid
AC	-	Activated Carbon
AH	-	Antioxidant
AIL	-	Acid Insoluble Lignin
A.M.U	-	Atom Mass Unit
ANOVA	-	Analysis of Variance
AP	-	Adequate Precision
APHA	-	American Public Health Association
ASL	-	Acid Soluble Lignin
ASTM	-	American Society for Testing and Material
ATCC	-	American Type Culture Collection
BBD	-	Box-Behnken Design
BHA	-	Butylated Hydroxyanisole
BHT	-	Butylated Hidroxytoluene
BPA	-	Baird-Parker Agar
C	-	Carbon
C-MWPA	-	Concentrated Ethyl Acetate Extract Microwave Pyrolygneous Acid
C-MPOB-PA	-	Concentrated Ethyl Acetate Extract Malaysian Palm Oil Board Pyrolygneous Acid
C ₁₂ H ₂₂ O ₁₁	-	Cellobiose
C ₂ H ₂	-	Acetylene
CH ₄	-	Methane
CO	-	Carbon Monoxide
CO ₂	-	Carbon Dioxide
CCD	-	Central Composite Design

CPA	-	Concentrated Pyroligneous Acid
CPAEA	-	Concentrated Pyroligneous Acid Ethyl Acetate
CRM	-	Coats-Redfern Method
CV	-	Coefficient Variation
db	-	dry basis
DIZ	-	Diameter Inhibition Zone
DCM	-	Dichloromethane
DF	-	Degree of Freedom
DNA	-	Deoxyribonucleic Acid
DP	-	Degree of Polymerization
DPPH	-	2,2-diphenyl-1-picrylhydrazyl
DOE	-	Design of Experiment
DTG	-	Derivative Thermogravimetric
EA	-	Ethyl Acetate
EC ₅₀	-	Half Maximal Effective Concentration
EDTA	-	Ethylenediaminetetraacetic acid
EFB	-	Empty Fruit Bunch
FeCl ₂	-	Iron (II) Chloride / Ferrous Chloride
FESEM	-	Field Emission Scanning Electron Microscope
FeSO ₄ ·7H ₂ O	-	Iron (II) Sulfate Heptahydrate
FFB	-	Fresh Fruit Bunch
FRAP	-	Ferric Reducing Antioxidant Power
GA	-	Gallic Acid
GAE	-	Gallic Acid Equivalents
GC-MS	-	Gas Chromatograph-Mass Spectroscopy
H	-	Hydrogen
H _p	-	Enthalpy of Pyrolysis
H ₂	-	Hydrogen Gas
H ₂ O ₂	-	Hydrogen Peroxide
H ₂ SO ₄	-	Sulfuric Acid
HBA	-	Hicrome™ Bacillus Agar
HPA	-	Hydrogen Peroxide Assay
HPLC	-	High Performance Liquid Chromatography
H ₃ PO ₄	-	Phosphoric acid
IC ₅₀	-	Half Maximal Inhibitory Concentration

ID	-	Inner Diameter
IKP	-	Isokinetic Point
KAS	-	Kissinger-Akahira-Sunose
KCE	-	Kinetic Correlation Effect
KFT	-	Karl Fischer Titration
LAP	-	Laboratory Analytical Procedure
LPM	-	Litre Per Minute
MBC	-	Minimum Bactericidal Concentration
MCA	-	Metal Chelating Activity
MIC	-	Minimum Inhibitory Concentration
MLC	-	Minimum Lethal Concentration
MPOB	-	Malaysian Palm Oil Board
MRSA	-	de Man, Ragosa, Sharpe Agar
MW	-	Microwave Heating
MWPA	-	Microwave-Assisted Pyrolygneous Acid
N	-	Nitrogen
NaHCO ₃	-	Sodium Bicarbonate
NIST	-	National Institute of Standards and Technology
NO	-	Nitric Oxide
O	-	Oxygen
O-CH ₃	-	Alkoxy
OD	-	Outer Diameter
OFW	-	Ozawa-Flynn-Wall
OH	-	Hydroxyl
OPF	-	Oil Palm Fiber
OPT	-	Oil Palm Trunk
PA	-	Pyrolygneous Acid
PAH	-	Polycyclic Aromatic Hydrocarbon
PBDEs	-	Polybrominated Diphenyl Ethers
PKS	-	Palm Kernel Shell
POME	-	Palm Oil Mill Effluent
R	-	Universal Gas constant
RPA	-	Raw Pyrolygneous Acid
ROS	-	Reactive Oxygen Species
RSM	-	Response Surface Methodology

RPA	-	Raw Pyroligneous Acid
S	-	Sulphur
SCA	-	Simmon Citrate Agar
SO ₂	-	Sulphur Dioxide
SRS	-	Sugar Recovery Standard
T	-	Absolute Temperature
TBHQ	-	Tertbutylhydroquinone
TCD	-	Thermal Conductivity Detector
TG	-	Thermogravimetric
TGA	-	Thermogravimetric Analysis
TPC	-	Total Phenolic Content
TPTZ	-	2,4,6-tripyridyl-striazine
UIRL	-	Universiti Industries Research Laboratory
UV-Vis	-	Ultra Violet Visible

LIST OF SYMBOLS

α	-	Conversion
A	-	Pre-exponential factor
β	-	Heating rate
cm	-	Centimeter
ΔG	-	Gibbs Free energy change
ΔH	-	Enthalpy change
ΔS	-	Entropy change
$^{\circ}\text{C}$	-	Degree celsius
$^{\circ}\text{C}/\text{min}$	-	Degree Celsius per minute
cells/mL	-	Cells per milliliter
dm^3	-	Cubic decimeter
$\text{dm}^3 \text{ dm}^{-3}$	-	Cubic decimeter per cubic decimeter
E_a	-	Activation energy
eV	-	Electron volt
E/g	-	Equivalent per gram
ϵ'	-	Dielectric constant
ϵ''	-	Dielectric loss
g	-	Gram
GHz	-	Gigahertz
h	-	Planck's constant
hr	-	Hour
J/mol	-	Joule per mol
k	-	Rate constant
K	-	Kelvin
Kg/dm^3	-	Kilogram per cubic decimeter
m	-	Meter

M	-	Molar
MJ kg ⁻¹	-	Mega Joules per kilogram
M ⁻¹ S ⁻¹	-	Per Molar per second
min	-	Minute
mg	-	Miligram
MHz	-	Megahertz
mL	-	Mililiter
mm	-	Milimeter
mM	-	Milimolar
mmol	-	Milimol
mg/ml	-	Miligram per mililiter
ml/min	-	Mililiter per minute
mg/L	-	Miligram per liter
mg/g	-	Miligram per gram
MJ/kg	-	Megajoules per kilogram
nm	-	Nanometer
%	-	Percent
ppm	-	Part per million
mt	-	Metric tonnes
>	-	More than
<	-	Less than
N	-	Avogadro's Number
R [•]	-	Radical Species
R ²	-	Correlation Coefficient
rpm	-	Revolution per minutes
T	-	Temperature
tha ⁻¹	-	Tonnes per hectare
t y ⁻¹	-	tonnes per year
μg	-	Microgram
μL	-	Microliter
μL/mL	-	Microliter per mililiter
μm	-	Micrometer
Vol. %	-	Volume percent
v/v/v	-	Volume per volume per volume
W	-	Watt

wt.%	-	Weight percent
wt./min	-	Weight percent per minute
w/v	-	Weight per volume

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	Location for OPF sampling	246
B	Analysis of lignin content	247
C	Proximate analysis of OPF	248
D	Ultimate Analysis of OPF	250
E	Standard calibration curve of Gallic acid and FeSO ₄ .7H ₂ O	251
F	Temperature profiles of microwave-assisted pyrolygneous acid for all standard runs	252
G	GC-MS ion chromatogram of optimized C-MWPA and C-MPOB	256
H	Two fold dilution table calculation for MIC and MBC	257
I	MIC observation for 0.5:1 and 1:1 of C-MPOB	258
J	Kinetic study analysis	260
K	Validation kinetic analysis of probable Reaction models	264
L	Kinetic correlation effect for OPF biomass and optimized C-MWPA	265

CHAPTER 1

INTRODUCTION

1.1 Research Background

In the last four decades, Malaysia has recorded unprecedented growth to emerge as the second largest producer as well as exporter of palm oil in the world where almost 5.4 million hectares of land were allocated for oil palm (*Elaeis guineensis*) plantation (Abas and Ani, 2014). It was estimated that every year Malaysia produces 22.1 million tonnes of palm oil from 37.2×10^6 tonnes of fresh fruit bunches compared to 15 million tonnes in 2005 (MPOB, 2015). This inevitably also produces huge amounts of biomass residues (yearly generation of around 100 million tonnes) and the trend is increasing annually by 5% (Abdullah and Sulaiman, 2013; Wafti *et al.*, 2017). This oil palm biomass residues includes empty fruit bunches (EFB), oil palm fiber (OPF), palm kernel shell (PKS), oil palm trunks (OPT) and palm oil mill effluent (POME) offers huge potential to be applied as raw materials to obtain valuable compounds based on its rich organic compositions (Wu *et al.*, 2007; Abas and Ani, 2014; Hosseini and Wahid, 2014).

Current management practice for oil palm residue, includes landfill and open burning, necessitates an improve approach as with time these biomass will contribute to uncontrolled released of greenhouse gases and global warming to the

environment (Hassan *et al.*, 2011). In addition, the open burning of biomass will lead to haze hazard, smoke as well as emission of toxic chemicals (Abdullah and Sulaiman, 2013). Due to this situation, proper disposal and management of oil palm biomass is necessary to minimize the environmental impact as well as maximizing utilization of these biomass into various value-added products such as biofuel, bio-oil, biochar, pyrolygneous acid and activated carbon that can be utilized in a wide range of applications. In general, biomass conversion technologies can be classified into three major processes namely thermochemical, physical and biochemical conversion (Figure 1.1).

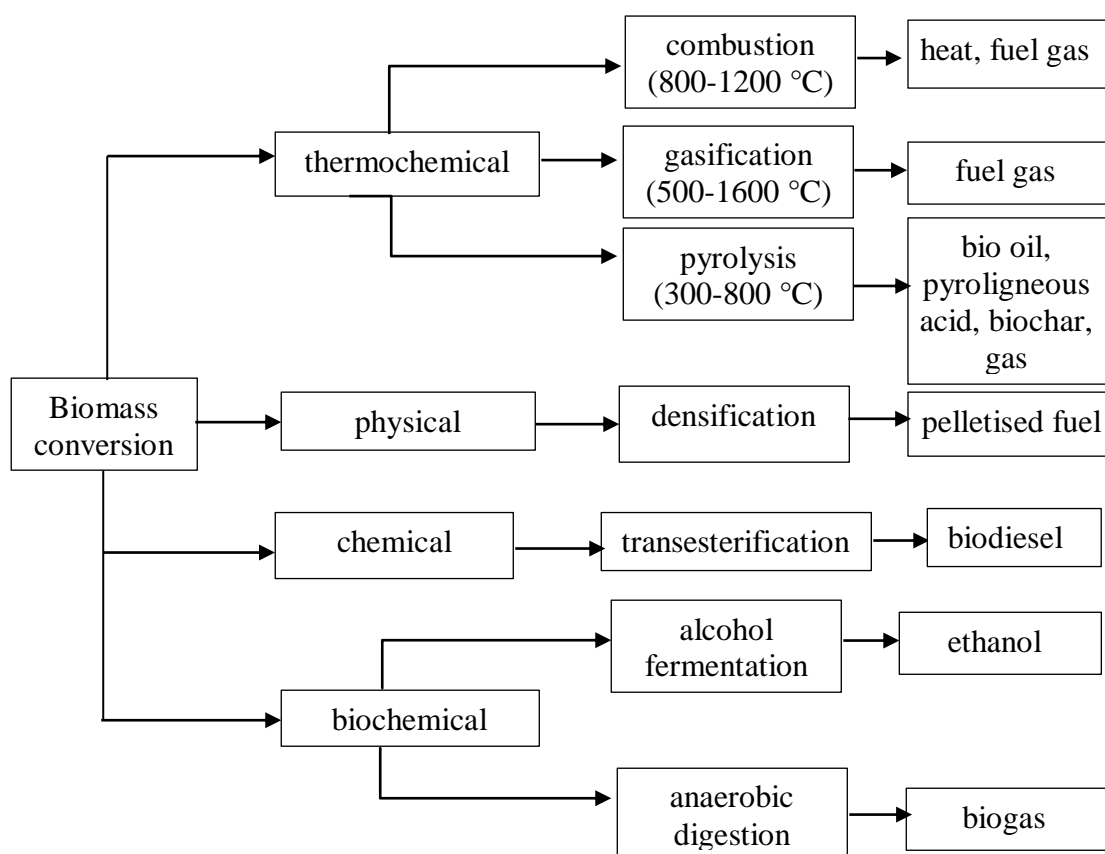


Figure 1.1 The main process of biomass conversion (Ani, 2012; Asomaning *et al.*, 2018)

Biochemical conversion involves the use of microorganisms whether as whole cells or extracted enzymes for the production of biogas and ethanol via anaerobic digestion and alcohol fermentation respectively. Thermochemical conversion has been acknowledged as the efficient process in handling various kind

of biomass and solid waste with a higher conversion rate in a rapid process time compared to others (Shakorfow, 2015) and it consist of combustion, gasification and pyrolysis. Thermochemical conversion is a proven technique both to dispose various types of biomass wastes as well as the production of valuable products such as bio-oil, pyrolygneous acid, biochar and fuel-gas (Ani, 2012; Goyal *et al.*, 2008; Lam and Chase, 2012). Amongst the available thermochemical conversion techniques, pyrolysis offers the advantage of being an environmental friendly method as well as an independent process to convert biomass into valuable chemical products (Ingole *et al.*, 2016; Czajczyńska *et al.*, 2017). Pyrolysis also offers simple operation, reasonable operating cost with lower range of the temperature required (350 to 800 °C) and produce less hazardous gas (CO₂, CO) relative to combustion (Shakorfow, 2015, Czajczyńska *et al.*, 2017). Apart from this, pyrolysis also acts as the initial step for all thermochemical processes that emits less air pollutants such as polybrominated diphenyl ethers (PBDEs) due to lower process temperature requirement. In contrast, gasification involved high energy consumption to maintain high process temperature required (Czajczyńska *et al.*, 2017; Yuan *et al.*, 2017). Table 1.1 highlights some of the characteristics of each processes:

Table 1.1: Characteristic comparison between gasification, combustion and pyrolysis (Shakorfow, 2015)

Characteristic	Gasification	Combustion	Pyrolysis
Process requirement	occurred in insufficient or partial air or oxygen or steam	occurred in sufficient oxygen or excess air	occurred in the absence of oxygen
Temperature	550-1600 °C	800 to 1200 °C	350 to 800 °C
Heat supply	allo or auto-thermal (internal heating)	exothermal	allo-thermal (external heating)
Carbon conversion	80-95 %	>99 %	≈ 75 %
Phase	gas	gas	solid, liquid, gas
Final product	fuel gases (CO ₂ , H ₂ O, N ₂) heat, tar and combustible gases (CO, H ₂ and CH ₄)	heat, fuel gas and gases as: CO ₂ , H ₂ O, N ₂ .	bio-oil, char, tar (liquid/vapour), CO ₂ , H ₂ O, and combustible gases
Reactivity of main product	stable, combustible	non-reactive	reactive, combustible

Pyrolysis can be defined as thermal decomposition of organic material in the absence of oxygen which is operating at medium temperature range normally from 350-550°C. Generally, it can be categorized into three types namely flash pyrolysis (favoring gas production), fast pyrolysis (principal product is liquid) and slow pyrolysis (applied for the production of char). The difference in pyrolysis types is determined mainly by two process parameters i.e. heating rate and residence time (Abubakar and Ani, 2013). Initial decomposition of waste material is around 120°C-200°C. During pyrolysis, decomposition of large molecule of biomass causing the breakdown of long chains of carbon, hydrogen and oxygen compounds into smaller molecules in the forms of condensable vapours (tars and oils), solid charcoal and non-condensable gaseous product (Abas and Ani, 2014; Yaman, 2004). Generally, pyrolysis of biomass consists of three main stages which includes; (i) initial evaporation of free moisture, (ii) primary decomposition and (iii) secondary reactions (oil cracking and repolymerisation). These stages are intermingled, with a possibility to observe their transitional behavior through thermal analysis (Kan *et al.*, 2016). In addition, the temperature of pyrolysis process can be adjusted in order to favor charcoal, pyrolysis liquid or gas production (Goyal *et al.*, 2008).

Pyroligneous acid (PA) is the aqueous liquid fraction that can be produced by condensing the vapor produced during pyrolysis of plant biomass in the absence of oxygen. It is also known as liquid smoke that has a distinctive smoky odor, reddish brown in color and acidic in nature (pH 2-3). Most of the studies carried out on PA production were using conventional heating methods that include charcoal kiln, jacket electric heater and furnace (rotating cone reactors, melting vessels, tabular reactor, blast furnaces). Amongst the disadvantages of conventional heating pyrolysis are slow reaction time, heat transfer resistance, inefficient heating due to heat losses to surrounding, lack of rapid heating, non-selective heating and damage to the reactor walls due to continuous electrical heating (Salema and Ani, 2011). Thus, microwave heating has promptly become an interesting alternative in the various industrial process in recent years. Microwave heating is widely used not only in analytical, organic and environmental chemistry but also in the pyrolysis of various kind of materials such as biomass, coal, oil shales and organic wastes (Domínguez *et al.*, 2006). Microwave heating offers many advantages over conventional heating including rapid internal heating, shorter processing time, lower

relative energy consumption, environmental friendliness, quick start-up, automated and volumetric heating. The application of microwave-assisted technology applied to the pyrolysis process not only contribute to the ability to enhance the reaction rates and rapid heating during pyrolysis, but also can improve the quality and properties of the product required and has been regarded as a way of cost-effective and operationally feasible processes (Menéndez *et al.*, 2010; Nomanbhay *et al.*, 2017).

Oil palm fiber (OPF) is one of the most abundant biomass produced during palm oil milling process. It is obtained after the pressing process of palm fruits for palm oil extraction. OPF can be converted into useful products for various applications such as in biofuel, bioethanol, biochemical, biocompost as well as biosugar. OPF is clean, non- carcinogenic, free from pesticides and soft parenchyma cell. Cellulose content has been observed as the principle component in chemical analysis of OPF since it plays an important role in the fiber's performance (Sreekala *et. al.*, 1997; Abdullah and Sulaiman, 2013). Thus, the analysis of the pyroligneous acid produced from OPF via microwave heating is required to get better understanding about the characteristic of the product, energy consumption, antioxidant and antimicrobial properties as well as kinetic analysis during the pyrolysis process.

1.2 Problem Statement

Numerous studies have investigated on the production of pyroligneous acid (PA) from various biomass feedstock by using conventional system such as the pyrolysis reactor and charcoal kiln. The quality of PA is based on the phenolic content. Production of PA by using conventional heating system is generally time consuming, requires high energy consumption, uneven heat distribution as well as generation of highly carcinogenic compounds such as polycyclic aromatic hydrocarbon (PAH). This situation leads to the production of low quality PA (low

phenolic contents). Microwave heating can be used to improve the quality of pyroligneous acid from its lower energy consumption, short pyrolysis time and better heat distribution with the expectation of high fraction of phenolic compounds produced with the absence of PAH. Amongst the available oil palm biomass in Malaysia, OPF offers an attractive alternative as feedstock for the production of PA based on its high lignin content. Nevertheless, to date, investigation on the production of PA from OPF via microwave-assisted heating in optimized condition is still very limited, hence is the focus of this study. The optimization process is important to determine optimum pyrolysis condition to obtain highest total phenolic content (TPC) as well as highest yield of PA. High phenolic content in PA would directly indicate high antioxidant and antimicrobial properties. Optimization would also lead to better understanding of the relationship between parameters verified (i.e holding time, final temperature and activated carbon loading) with the responses (TPC and yield of PA). Kinetic analysis is important for detail understanding on the pyrolysis temperature range and the behavior of lignocellulosic material degradation (cellulose, hemicellulose and lignin) during pyrolysis. Kinetic parameters such as activation energy (E_a), pre-exponential factors, (A) and reaction model are valuable information for any scaling up attempts notably for design of equipment and process optimization condition for yield maximization.

1.3 Research Objectives

The objective of this study were as follows:

- i) To optimize the production of total phenolic content (TPC) and liquid yield of microwave-assisted pyroligneous acid from OPF.
- ii) To evaluate the efficiency of optimized microwave-assisted pyroligneous acid as antioxidant and antimicrobial agents.
- iii) To elucidate the kinetic study analysis of OPF biomass and optimized microwave-assisted pyroligneous acid for details understanding on the

energy requirement and mechanism behavior of the material degradation during pyrolysis process.

1.4 Scope of Study

- i) The pyroligneous acid was synthesized from OPF via microwave-assisted pyrolysis heating.
- ii) The activated carbon was used as microwave absorber to produce pyroligneous acid from OPF via microwave-assisted pyrolysis heating.
- iii) Three parameters were used during the optimization study namely holding time (15-30 minutes), final temperature (400-600 °C) and amount of activated carbon loading (50-100 g).
- iv) The Central Composite Design (CCD) approach via response surface methodology (RSM) was used for the optimization process on the TPC as well as liquid yield of microwave-assisted pyroligneous acid (MWPA).
- v) The efficiency of optimized concentrated microwave-assisted pyroligneous acid extracted with ethyl acetate (C-MWPA) was evaluated for antioxidant and antimicrobial properties. Ethyl acetate (99.5%, AnalaR grade) was used due to its less toxicity and higher capacity to extract phenolic compound in MWPA.
- vi) Kinetic study analysis were based on thermogravimetric analysis (TGA) data using three kinetics models approach namely Ozawa-Flynn Wall (OFW) Kissinger-Akahira-Sunose (KAS) and Coats-Redfern method (CRM).
- vii) The kinetic model validation of OPF biomass and optimized C-MWPA were analyzed using 13 types of reaction models from CRM.

1.5 Significance of Study

The establishment on the production of pyroligneous acid containing highest amount of total phenolic content and highest yield in optimized microwave-assisted pyrolysis process can lead to larger application such as at pilot scale level. Process optimization would allow significant energy and time saving. The kinetic study analysis assist in the understanding of pyrolysis temperature range and the behavior of biomass degradation during pyrolysis. The combination of model free method (KAS and OFW) with the model fitting method (CRM) significantly contribute to the ability to reveal the complexity of the process and to find the best fitted kinetic reaction model of the material involved during pyrolysis process. Moreover, it also contribute to the minimization in the volumes of oil palm biomass waste that needs to be transported to the plantation area from the mills. This directly contributes to the reduction in transportation cost as well as reduction in environmental pollution from degradation of these biomass.

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