

CHARACTERIZATION AND KINETICS STUDY OF
ACTIVATED COCONUT SHELLS, COW BONES
AND ZEOLITE BASED ADSORBENT FOR POME
TREATMENT

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PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

A thesis submitted in
Fulfillment of the requirement for the award of the
Doctorate Degree of Philosophy of Civil Engineering

Faculty of Civil and Environmental Engineering
Universiti Tun Hussein Onn Malaysia

JANUARY 2018

DEDICATION

This project is dedicated to Almighty Allah, the maker of all things and to my family especially my parents Mr. and Mrs. Adeleke who have being the pillar of my life from cradle.



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ACKNOWLEDGEMENT

I wish to express my profound gratitude to Almighty Allah who has made my studies to be possible overseas. I would like to extend my gratitude to my supervisor Prof. Ab Aziz Abdul Latiff for his guide, support, motivation and mentorship and for giving me the platform and opportunity to pursue this research. I cannot quantify my gratitude Prof, may Allah reward you abundantly. I am also thankful to my Co- Supervisor, Assoc. Prof Zawawi Daud for his assistance and encouragement and other members on my research team.

I wish to acknowledge both the academic and non-academic staff of the Faculty, my sincere thanks go to my Head of Department, Assoc. Prof. Mohammed Adib Mohd Razi, Deputy Dean of research and innovation, Associate Professor Norzila Othman, Director of micropollutant research center (MPRC) Associate Professor Radin Maya Mohammed Saphira and all the staff of water and environmental engineering.

I also wish to extend my gratitude to the science officers and colleagues; Falilah Mat Daud, Mohammed Nda, Dr Arif Rosli, Mahmood Hijab, Vicky Kumar, Mohammed Kabir and others. Thank you and God bless you all. I appreciate the assistance and patience granted to me by the laboratory technician of MPRC to use the laboratory facilities even beyond the official hours. I will not forget to thank the cleaners who made the workstation to be habitable throughout my study period.

My special gratitude goes to my family members, my father, my mother, siblings and my fiancée (AbdulKareem Robiah). Thank you for your support. Your love and care is well acknowledged. I really appreciate all the kind gestures, motivation and prayers. May Allah reward you all with long life and prosperity.

Finally, I would like to express my gratitude to the office of research, innovation and commercialization center (ORICC) Universiti Tun Hussein Onn Malaysia for making my study possible through the support with the research grant (U272).



ABSTRACT

Palm oil mill effluent (POME) is a high strength agro-allied wastewater containing both organic pollutants and heavy metals. The discharge of POME into the environment without adequate treatment contributes to diseases affecting humans and aquatic lives. However, there is the necessity to reduce the pollutants to a very low level of discharge to reduce the impact of the toxic effect of the pollutants on the environment and the aquatic population. The conventional approach for the treatment of POME is expensive compared to the method of adsorption. The method of adsorption has shown to be cost and time effective for research. Thus, the objective of this study was to prepare composite adsorbent from activated coconut shell carbon (ACSC), activated cow bone powder (ACBP) and zeolite for the treatment of POME using the optimum particle size obtained in a batch adsorption study. The characterization of the ACSC, ACBP and zeolite was obtained using pendant drop contact angle experiment. The result illustrated that the contact angles of the ACSC, ACBP and zeolite respectively were 105.20°, 95.70° and 25. 20°. The result of the contact angles showed that activated coconut shell carbon and activated cow bone powder were hydrophobic materials while the zeolite was hydrophilic. The investigation of the chemical composition of the materials using energy dispersive x-ray (EDX) indicated that the major elements of both ACSC and ACBP were predominantly C, Ca²⁺ and O and Si for zeolite and C, Si, O, Na, Mg, Ca and P as the major elements on the surface of the composite while the XRF showed that the composite contained CaO and SiO₂ as the major compounds. The point of zero charge (pH_{pzc}) of 5.28 achieved showed that the composite contained acidic surface which influenced cationic exchange in the supernatant and the surface of the composite. The CEC after adsorption was observed as 0.8918±0.0669 meq/g. The optimal batch adsorption of COD and NH₃-N was obtained at under fixed condition of pH 7, 105 minutes contact time at 150 rpm shaking speed and 150 µm particle size for ACSC,



ACBP and zeolite. The prepared composite adsorbent contained functional groups of CH, C=C, C-O-C, OH using the Fourier transform irradiation (FT-IR) analysis. The optimal operation parameters of the adsorption process for the reduction of COD and NH₃-N using the central composite design (RSM) was recorded at pH 10, 50 rpm of shaking speed for 2 h and by using 3 mm of composite particle size and 125 gL⁻¹ of the adsorbent at initial concentration of POME of 1 ml per 500 ml volumetric flask. The results revealed that the investigated factors evidently induced the reduction of the parameters. The experimental data of COD, NH₃-N, Cd, Fe and Pb from the batch study were fitted to the isotherm and kinetic models. The result of the isotherm study fitted best to the Langmuir equation model for COD, NH₃-N, Fe and Cd which indicated that the adsorption of the pollutants from the supernatant was favourable on a mono layer surface. The Freundlich isotherm fitted experimental data better than the Langmuir and the Temkin isotherm for Pb which showed that the adsorption process was effective on a heterogeneous surface. The investigation showed that the uptakes of COD, NH₃-N, Cd, Fe and Pb from the experimental data were fitted to the pseudo-second order kinetic model which implied that the process of adsorption was by chemisorption. Furthermore, the fixed bed packed composite adsorption was conducted and the experimental data were fitted to Thomas and Adams-Bohart model. The model parameters were obtained from the breakthrough curves, the effective adsorption of COD was obtained at lower flow rate influent concentration. Desorption of the spent adsorbent was suitably conducted using 0.5M NaOH and breakthrough was obtained at longer retention time. It can be concluded that a mesoporous and granular composite adsorbent was effective for the treatment of both organic and heavy metal pollutants. It was observed that both batch isotherm and kinetic study can be effectively applied for the treatment of POME using the composite adsorbent, although the effectiveness of the batch adsorption study showed to be more suitable than the fixed bed continuous column for the removal of pollutants of POME. However, due to the potential of the composite adsorbent for the treatment of POME, the treatment efficiency of the adsorbent has shown that the composite have the potential to be used for the treatment of pollutants of high strength wastewater.



ABSTRAK

Efluen kilang minyak kelapa sawit (POME) adalah air sisa agro-pertanian yang mengandungi bahan pencemar organik dan logam berat. Pelepasan POME ke dalam persekitaran tanpa rawatan yang mencukupi menyumbang kepada penyakit yang memberi kesan kepada manusia dan kehidupan akuatik. Walau bagaimanapun, terdapat keperluan untuk mengurangkan pencemaran kepada tahap pelepasan yang sangat rendah untuk meminimumkan kesan pencemar toksik terhadap alam sekitar dan populasi akuatik. Pendekatan konvensional untuk rawatan POME adalah mahal berbanding kaedah penjerapan. Kaedah penjerapan telah menunjukkan kos dan masa yang berkesan untuk penyelidikan. Oleh itu, objektif kajian ini adalah untuk menyediakan penjerap komposit daripada karbon teraktif tempurung kelapa (ACSC), serbuk tulang lembu teraktif (ACBP) dan zeolit untuk rawatan POME menggunakan saiz partikel optimum yang diperolehi dalam ujikaji penjerapan kelompok. Pencirian ACSC, ACBP dan zeolit diperolehi dengan menggunakan ujikaji sudut sentuhan titis loket. Hasil keputusan menggambarkan bahawa sudut sentuhan ACSC, ACBP dan zeolit masing-masing adalah 105.20° , 95.70° dan 25.20° . Hasil daripada sudut sentuhan menunjukkan bahawa karbon teraktif tempurung kelapa dan serbuk tulang lembu teraktif adalah daripada bahan hidrofobik manakala zeolit adalah daripada bahan hidrofilik. Penyiasatan komposisi bahan kimia yang menggunakan sinaran-x penyebaran tenaga (EDX) menunjukkan bahawa unsur-unsur utama ACSC dan ACBP adalah terutamanya C, Ca^{2+} dan O dan Si untuk zeolit dan C, Si, O, Na, Mg, Ca dan P sebagai unsur utama pada permukaan komposit manakala XRF menunjukkan bahawa komposit mengandungi CaO dan SiO_2 sebagai sebatian utama. Titik caj sifar (pH_{pzc}) adalah 5.28 telah dicapai menunjukkan bahawa komposit mengandungi permukaan berasid yang mempengaruhi pertukaran kationik dalam supernatan dan permukaan komposit. CEC selepas penjerapan diperhatikan sebagai 0.8918 ± 0.0669 meq/g. Penjerapan kelompok optimum COD dan $\text{NH}_3\text{-N}$ didapati dalam keadaan tetap iaitu pH 7, 105 minit masa sentuhan pada kelajuan goncangan 150 rpm dan saiz partikel $150\mu\text{m}$ untuk ACSC, ACBP dan zeolit. Penjerap komposit yang disediakan



mengandung kumpulan berfungsi CH, C=C, C-O-C, OH menggunakan analisis radiasi transformasi Fourier (FT-IR). Parameter optimum operasi bekerja bagi proses penjerapan untuk penurunan COD dan NH₃-N menggunakan reka bentuk komposit pusat (RSM) direkodkan pada pH 10, kelajuan goncangan 50 rpm selama 2 jam dan menggunakan 3 mm saiz partikel komposit dan 125 gL⁻¹ dari penjerap pada kepekatan awal POME 1 ml terhadap 500 ml kelalang volumetrik. Hasil keputusan didapati bahawa faktor-faktor yang dikaji secara jelasnya mendorong terhadap penurunan parameter. Data eksperimen COD, NH₃-N, Cd, Fe dan Pb dari ujikaji kelompok dipadankan dengan model isoterma dan kinetik. Hasil kajian padanan isoterma terbaik adalah model persamaan Langmuir untuk COD, NH₃-N, Fe dan Cd yang menunjukkan bahawa penjerapan bahan pencemar dari supernatan adalah digemari pada permukaan lapisan tunggal. Data eksperimen daripada isoterma Freundlich yang dipadankan adalah lebih baik daripada isoterma Langmuir dan Temkin untuk Pb yang menunjukkan bahawa proses penjerapan berkesan pada permukaan yang heterogen. Penyiasatan didapati menunjukkan bahawa pengambilan COD, NH₃-N, Cd, Fe dan Pb daripada data eksperimen telah dipadankan dengan model kinetik pseudo-tertib kedua yang menunjukkan bahawa proses penjerapan adalah secara penjerapan kimia. Seterusnya, penjerapan komposit lapisan tetap yang telah dijalankan dan data eksperimen berikut telah dipadankan dengan model Thomas dan Adams-Bohart. Parameter model telah diperolehi daripada lengkung bulus, penjerapan COD yang berkesan ditunjukkan melalui kadar aliran yang lebih rendah. Penjerapan semula penjerap telah dilakukan dengan menggunakan 0.5M NaOH dan bulus didapati pada masa tahanan yang lebih lama. Secara kesimpulannya didapati bahawa penjerap komposit mesolintang dan butiran adalah berkesan untuk merawat kedua-dua bahan pencemar organik dan logam berat. Telah diperhatikan bahawa kajian isoterma dan kinetik kedua-duanya secara berkesan boleh digunakan untuk rawatan POME dengan menggunakan penjerap komposit, walaupun keberkesanan ujikaji penjerapan kelompok menunjukkan ianya lebih sesuai daripada turus penjerapan lapisan tetap untuk penyingkiran bahan pencemar POME. Walau bagaimanapun, disebabkan oleh potensi penjerap komposit untuk rawatan POME, kecekapan rawatan penjerap telah menunjukkan bahawa penjerap komposit mempunyai potensi untuk digunapakai dalam rawatan pencemaran air sisa berkekuatan tinggi.



TABLE OF CONTENTS

DECLARATION	ii
DEDICATION	iii
ACKNOWLEDGEMENT	iv
ABSTRACT	v
ABSTRAK	vii
TABLE OF CONTENTS	ix
LIST OF TABLES	xv
LIST OF FIGURES	xviii
LIST OF APPENDICES	xxv
LIST OF PUBLICATIONS	xxvi
LIST OF AWARDS	xxviii

CHAPTER 1 INTRODUCTION

1.1	Background of Study	1
1.2	Problem Statement	3
1.3	Objectives of the Study	6
1.4	Hypothesis	7
1.5	Scope of Work	7
1.6	Significance of the Study	8
1.7	Thesis Organization	9

CHAPTER 2 LITERATURE REVIEW

2.1	Introduction	10
2.2	Palm Oil Processing and Production	11
2.3	Palm Oil Mill Effluent	12
2.4	Trends in POME Treatment Methods	15
	2.4.1 Conventional Treatment Method	16
	2.4.2 Physical and Chemical Processes	22
2.5	Adsorption	30
	2.5.1 Physiosorption	31
	2.5.2 Chemisorption	31
2.5.3	Wetting and Fluid Adsorption on Adsorbent Surfaces	33
2.5.4	Surface Tension and Contact Angles	33
2.6	Hydrophobicity and Hydrophilicity of Adsorbent Material	34
	2.6.1 Hydrophobicity of Adsorbent Material	34
	2.6.2 Hydrophilicity of Adsorbent Materials	35
2.7	Treatment of POME Using Adsorption	36
2.8	Adsorbent Materials	40
	2.8.1 Activated Carbon Adsorbent	40
	2.8.2 Application of Locally Sourced Adsorbents Materials	42
	2.8.2.1 Coconut Shell Adsorbent	44
	2.8.2.2 Bone Activated Carbon Adsorbent	45
	2.8.2.3 Zeolite Adsorbent	46
2.8.3	Composite Adsorbent	47
2.8.4	Optimization of Operating Conditions for the Adsorption Process	48
2.9	Adsorption Isotherm and Mechanism	50
2.10	Kinetic Adsorption Studies	57
	2.10.1 Pseudo-First Order Equation	58
	2.10.2 Pseudo- Second Order Equation	59
2.11	Fixed Bed Adsorption	63
	2.11.1 Principle of Column Adsorption	64
	2.11.2 Breakthrough Curve Model	65
	2.11.2.1 Thomas Model	65



2.11.2.2	The Yoon- Nelson Model	66
2.11.2.3	Adam-Bohart Model	66
2.12	Key Findings of Literature Review	67

CHAPTER 3 METHODOLOGY

3.1	Introduction	69
3.2	Materials	71
3.2.1	Reagents and Chemicals	71
3.3	Preparation of the Adsorbate Solution	72
3.3.1	Standard Solution for Heavy Metals	73
3.3.2	Preparation of Calibration Curve	73
3.4	Preparation of Composite Materials	74
3.4.1	Experimental Analysis of the Starting Adsorbents Materials	75
3.4.2	Experimental Analysis for the Activated Coconut Shell Carbon	75
3.4.3	Experimental Analysis for the Preparation of Activated Cow Bone Powder	76
3.4.4	Experimental Analysis of Zeolite	77
3.5	Surface Characterization of Adsorbent	78
3.5.1	Physical Characterization	78
3.5.1.1	Surface Tension	80
3.5.1.2	Bulk Density	81
3.5.1.3	Porosity Volume	82
3.5.1.4	Morphology Study	82
3.5.1.5	BET Surface Area	83
3.5.2	Chemical Characterization	81
3.5.2.1	Chemical Composition	81
3.5.2.2	Functional Group	82
3.5.2.3	Point of Zero Charge	82
3.5.2.4	Cation Exchange Capacity	82
3.6	Optimization of Process Parameters	83
3.6.1	Batch Adsorption Experiment	84



3.6.1.1	Linear Optimization and the Preparation of Composite	85
3.6.2	Optimization of the Process Parameters using Central Composite Design	90
3.6.2.1	Experimental Design	91
3.7	Adsorption Isotherm Studies	91
3.8	Batch Adsorption Kinetic Studies	91
3.9	Procedure for Fixed Bed Adsorption	92
3.10	Regeneration of Spent Composite Adsorbent	94

CHAPTER 4 RESULTS AND DISCUSSION

4.1	Introduction	95
4.2	Characterization of POME	95
4.3	Characterization of Adsorbents	98
4.3.1	Physical Characterization of the Adsorbents	98
4.3.1.1	Contact Angle of Adsorbent	98
4.3.1.2	Bulk Density of Adsorbent	101
4.3.1.3	Surface Morphology	102
4.3.2	Chemical Characterization of the Adsorbent	104
4.4	Preparation of the Composite Adsorbent	104
4.4.1	Linear Optimization of the Single Adsorbents	105
4.4.2	Optimization of Process Parameters of the Composite	116
4.4.2.1	Development of Regression Model for the Response Parameters on the Composite.	121
4.4.2.2	Surface Morphology of Composite	128
4.4.2.3	BET Surface Area of Composite	129
4.4.2.4	EDX Analysis of Composite	130
4.4.2.5	Point of Zero Charge of Composite	134
4.4.2.6	Cationic Exchange Capacity of Adsorbent	135
4.4.2.7	FTIR Analysis of Composite	137
4.5	Batch Adsorption Studies	139



4.5.1	Adsorption Isotherms	141
4.5.1.1	Batch Adsorption Isotherm of COD	142
4.5.1.2	Batch Adsorption Isotherm of NH ₃ -N	145
4.5.1.3	Batch Adsorption Isotherm of Cadmium	148
4.5.1.4	Batch Adsorption Isotherm of Lead	150
4.5.1.5	Batch Adsorption Isotherm of Iron	152
4.5.2	Batch Kinetic Studies	154
4.5.2.1	Batch Kinetic Study of COD on Composite	155
4.5.2.2	Batch Kinetic Study of NH ₃ -N on Composite	157
4.5.2.3	Batch Kinetic Study of Cadmium on Composite	159
4.5.2.4	Batch Kinetic Study of lead on Composite	161
4.5.2.5	Batch Kinetic Study of Iron on Composite	162
4.6	Fixed Bed Adsorption Studies	165
4.6.1	Introduction	165
4.6.1.1	Fixed Bed Column Studies	167
4.6.1.2	Effect of the Influent Flow Rate	167
4.6.2	Mechanism of the Packed Column Adsorption	169
4.6.2.1	Application of Thomas Model	171
4.6.2.2	Application of Adam-Bohart Model	171
4.7	Regeneration/ Desorption of Spent Composite	172

CHAPTER 5 CONCLUSION AND RECOMMENDATION

5.1	Conclusion	176
5.2	Recommendation	179

REFERENCES

APPENDIX A

215

APPENDIX B

221

VITAE



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

LIST OF TABLES

2.1	Characteristic of palm oil mill effluent (POME)	14
2.2	(a) Prevailing effluent discharge standard for crude palm oil mills	15
2.2	(b) Environmental Quality Act 1974 for POME Discharged	15
2.3	Treatment of POME using the anaerobic system	17
2.4	Physical-Chemical method of treatment of POME	23
2.5	Adsorption of POME	38
2.6	Optimum mixing ratio of hydrophobic and hydrophilic adsorbent	49
2.7	Separator factor	53
2.8	Adsorption isotherms of heavy metals and organic pollutants	55
2.9	Adsorption kinetics of pollutants onto the adsorbent	61
3.1	(a) List of reagents and chemicals	71
3.1	(b) POME characteristics and methods	72
3.2	Optimum ratio of the hydrophobic-hydrophilic ratio by volume of the adsorbent	86
3.3	Adsorbent-Binder Ratio	86
3.4	Experimental design for the optimization of the response factors	91
3.5	Design of Fixed Bed Column	92
4.1	(a) Physico-chemical characteristics of raw POME (N=3)	97
4.1	(b) Heavy Metal characteristics of raw POME (N=3)	97
4.2	Bulk densities of starting materials	102
4.3	(a): Batch Adsorption of single adsorbent of different particle sizes for COD removal	107
	(b) Batch Adsorption of single adsorbent of different particle sizes for NH ₃ -N removal	108
4.4	(a) Linear Optimization of Hydrophobic adsorbent	111

4.4	(b) Linear Optimization of Hydrophilic adsorbent	112
4.5	Linear optimization of Hydrophobic-hydrophilic adsorbent	114
4.6	Optimum mixing ratio of adsorbents	115
4.7	(a) Batch Adsorption of COD from the operating parameters of CCD	117
4.7	(b) Batch Adsorption of NH ₃ -N from the operating parameters of CCD	119
4.8	Regression Coefficient and their Significance of the Linear, Quadratic and Cubic Model for the Reduction of COD and NH ₃ -N from POME by using Central Composite Design	121
4.9	Analysis of the variance (ANOVA) of the response surface quadratic model for the reduction of COD and NH ₃ -N from POME by natural composite	125
4.10	Specific surface area and pore size distribution of composite	130
4.11	(a) Element composition of composite adsorbent	131
4.11	(b) Chemical Composition of the Composite Adsorbent	132
4.12	Cationic Exchange Capacity of Adsorbents	136
4.13	FT-IR Spectra absorption peak frequencies and the corresponding functional groups on the composite before and after adsorption process conducted at the optimal conditions	139
4.14	Batch adsorption isotherm of COD on composite	145
4.15	Batch adsorption isotherm of NH ₃ -N on composite	147
4.16	Batch adsorption isotherm of Cd on composite	149
4.17	Batch adsorption isotherm of Pb on composite	151
4.18	Batch adsorption isotherm of Fe on composite	153
4.19	Batch Kinetic Study of COD on composite	156
4.20	Batch Kinetic Study of NH ₃ -N on composite	158
4.21	Batch Kinetic Study of Cd on composite	160
4.22	Batch Kinetic Study of Pb on composite	162
4.23	Batch Kinetic Study of Fe on composite	163
4.24	Batch isotherm study of solutes on composite	165
4.25	Batch kinetic study of solutes on composite	165
4.26	Fixed bed adsorption of packed composite	167



4.27	Fixed bed desorption using eluting agent at optimum inflow rate	172
4.28	Fixed bed adsorption on regenerated composite at optimum flow rate	174



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

LIST OF FIGURES

2.1	Palm oil mill processing plant	13
3.1	Frame Work of Research Activities	70
3.2	Activated coconut shell carbon	75
3.3	Preparation of activated cow bone powder	77
3.4	Zeolite	77
3.5	Process of Preparation of composite	88
3.6	Preparation of composite	90
3.7	Diagram of the Column for Fixed Bed Adsorption	93
4.1	(a) Contact angle of activated coconut shell carbon	100
	(b) Contact angle of activated cow bone powder	100
	(c) Contact angle of zeolite	101
4.2	(a) Surface morphology of the activated coconut shell carbon	103
	(b) Surface morphology of activated cow bone powder	103
	(c) Surface morphology of zeolite	103
4.3	(a) Batch Adsorption of COD	106
	(b) Batch Adsorption of NH ₃ -N	109
4.4	(a) Optimization of hydrophobic adsorbents	110
	(b) Optimization of hydrophilic adsorbent	110
4.5	Optimization of hydrophobic-hydrophilic ratio	113
4.6	Optimization of the adsorbent-binder ratio	115
4.7 (a)	(1) Interaction between pH and initial concentration of composite	



	(2) Interaction between shaking speed and time	127
	(b) Interaction between time and adsorbent dosage	127
4.7 (b)	(1) Interaction between shaking speed and time	128
	(2) Interaction between time and adsorbent dosage	128
4.8	(a) SEM micrograph of the composite before treatment	128
	(b) Optimization of hydrophilic adsorbent	130
4.9	(a) Chemical composition of the composite before adsorption	132
	(b) SEM micrograph of the composite after treatment	134
4.10	Point of zero charge of the composite adsorbent	135
4.11	FT-IR analysis of the composite before and after treatment	137
4.12	Batch adsorption of COD removal on composite	140
4.13	Batch adsorption of $\text{NH}_3\text{-N}$ on composite	140
4.14	Linear regression of Langmuir isotherm for COD on composite	143
4.15	Linear regression of Freundlich for COD on composite	144
4.16	Linear regression of Temkin for COD on composite	144
4.17	Linear regression of Langmuir isotherm on $\text{NH}_3\text{-N}$	145
4.18	Linear regression of Freundlich Isotherm on $\text{NH}_3\text{-N}$	146
4.19	Linear regression of Temkin Isotherm on $\text{NH}_3\text{-N}$	146
4.20	Linear regression of Langmuir Isotherm on Cadmium	147
4.21	Linear regression of Freundlich on Cadmium	148
4.22	Linear regression of Temkin on Cadmium	148
4.23	Linear regression of Langmuir Isotherm on Lead	149
4.24	Linear regression of Freundlich Isotherm on Lead	150
4.25	Linear regression of Temkin isotherm on Lead	150
4.26	Linear regression of Langmuir isotherm on iron	151
4.27	Linear regression of Freundlich isotherm on iron	152
4.28	Linear regression of Temkin isotherm on iron	152
4.29	Linear regression of first order kinetic on COD	155
4.30	Linear regression of pseudo second order on COD	155
4.31	Linear regression of pseudo first order on $\text{NH}_3\text{-N}$	157
4.32	Linear regression of pseudo second order on $\text{NH}_3\text{-N}$	157
4.33	Linear regression pseudo first order kinetic on cadmium	159



4.34	Linear regression of pseudo second order kinetic on cadmium	159
4.35	Linear regression of pseudo first order on lead	160
4.36	Linear regression of pseudo second order kinetic on lead	161
4.37	Linear regression of pseudo first order on iron	162
4.38	Linear regression of pseudo second order on iron	163
4.39	Ion exchange of pollutants and elements on the functional group on the active surface of the composite	164
4.40	Break through curve at different flow rates influent concentration	168
4.41	Experimental data fitted to fixed bed Thomas model	170
4.42	Experimental data fitted to Adam-Bohart Model	171
4.43	Desorption of COD on spent adsorbent	173
4.44	Break through curve after regeneration of composite	173



LIST OF SYMBOLS AND ABBREVIATIONS

A	Binding surface area
B	Temkin constant
B	Blank
C _e	Final equilibrium concentration
C _o	Initial concentration
CO	Cobalt
C _v	Crystal violet
Cu	Copper
Cr	Chromium
Cd	Cadmium
C _T	Outlet concentration
D	Dilution factor
Fe	Iron
H	Initial rate of adsorption
K	Kinetic constant
K _f	Adsorbent adsorbed per unit equilibrium
K _i	Intraparticle diffusion rate constant
K _L	Langmuir constant
K _T	Temkin constant
K _{TH}	Thomas rate constant
K _{yn}	Rate constant
L	Liter
Lb	Pound
Ft	Feet
M	Mass
mg/L	Milligram per liter
Mn	Manganese
N	Total available sites
N	Freundlich slope
Ni	Nickel
N _s	Number of sites occupied



P	Pressure
P°	Vapour pressure
Q	Flow rate
q_e	Quantity of the adsorbed per unit mass of adsorbent
q_0	Maximum solid phase concentration of the solute
q_t	Amount of the adsorbed per unit time
R	Gas constant
R_L	Separation coefficient
T	Temperature
S	Sample
U	Speed of gas out
V	Volume of solution
V_{eff}	Volume of effluent
V_{ϵ}	Volume porosity
X	Amount of adsorbent in the column
W	Weight of adsorbent
Z	Column bed height
Λ	Time required for breakthrough at 50%
>	Greater than
<	Less than
°	Degree
AAS	Atomic analyst spectrometer
AF	Activated carbon
ACBP	Activated cow bone powder
ACSC	Activated coconut shell carbon
AF	Anaerobic filter
ASB	Activated sludge biomass
ASBR	Anaerobic sequencing batch reactor
ATA	Attapulgate composite
BDDT	Brunauer, Deming, Deming Teller
BDOC	Biodegradable organic compound
BET	Brunauer Emmet and Teller
BOD	Biochemical oxygen demand
BF	Basil Fuchsin
CA	Catalytic activity
CBP	Cow bone powder



CCD	Central composite design
COD	Chemical oxygen demand
CPW	Crude petroleum wastes
CR	Congo red
CV	Crystal violet
DOE	Department of Environment
DKR	Dubinini-Kagener Radushkevich
EBCT	Empty bed contact time
EDX	Energy dispersive x-ray
EF	Electron –Fenton process
EFB	Empty fruit bunches
EPA	Environmental protection agency
FAS	Ferrous ammonium sulphate
FO	Forward osmosis
FTIR	Fourier transform irradiation
GAC	Granular activated carbon
GCF	Global contamination factor
HAP	Hydroxyapatite
ICF	Individual contamination factor
ICP-MS	Inductively coupled plasma mass spectrometry
INWQS	Interim water quality standard
MAS	Membrane anaerobic system
MB	Methylene blue
MF	Micro filtration
MO	Methyl orange
MSDS	Material safety and data sheet
MTZ	Mass transfer zone
NF	Nano filtration
OLR	Organic loading rate
OPC	Ordinary Portland cement
OPF	Oil palm fronds
OPT	Oil palm trunk
PAC	Powdered activated carbon
PCB	Poly chlorinated biphenyls
POME	Palm oil mill effluent
POMS	Palm oil mill sludge
POFA	Palm oil fuel ash
PPB	Part per million
PPF	Palm processed fibers
PSAC	Palm shell activated carbon
PVA	Polyvinyl alcohol



RO	Reverse osmosis
RSM	Response surface methodology
SEM	Scanning electron microscopy
SS	Suspended solids
SWOT	Strength, opportunity, weakness and threat
TN	Total nitrogen
TOC	Total organic carbon
TS	Total solids
TSS	Total suspended solids
UASB	Up-flow anaerobic sludge blanket
UASFF	Up flow anaerobic sludge fixed film
UF	Ultra filtration
VSS	Volatile suspended solids
WAS	Waste activated sludge
WHO	World Health Organization



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	Characterization of Adsorbents	
	No table of figures entries found.	



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

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