

TEXTILE WASTEWATER TREATMENT USING MAGNETIC POWDER
ACTIVATED CARBON BIOGRANULES

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ABSTRACT

Biogranulation technology is novel in the field of biological wastewater treatment with high removal potential as well as providing economical and technical advantages. This technology has been widely tested in the degradation of various types of wastewater owing to its unique sludge properties and high biodegradability potential. Despite its unique characteristics, the major drawback of biogranulation is the long start-up period. This study investigated the possibility of developing biogranules with magnetic powder activated carbon (MPAC) in treating synthetic textile wastewater. This study was aimed at enhancing biogranules development process with better characteristics and high removal performance. At early stage of this study, the effects of magnetic field and MPAC on the initial process development of biogranules were studied using one factor at a time (OFAT) and response surface methodology (RSM). The cultivation of biogranules was then investigated using two laboratory scale sequencing batch reactors (SBR) under intermittent anaerobic and aerobic conditions. Reactor R1 acted as a control system while reactor R2 was added with MPAC. The reactors were designed with 3 L of total working volume and operated at 50% volumetric exchange rate. These biogranules were cultivated with a mixture of textile mill and municipal wastewater sludge. The systems were fed with synthetic textile wastewater. Removal performances, structural aspects and formation of MPAC biogranules were examined based on physical, biological and chemical properties. Batch test results showed that static magnetic field induction and MPAC gave significant positive effect on improving the initial biogranulation process. After 60 days of development stage in the SBR system, the average size of the biogranules increased, reaching $2.0 \text{ mm} \pm 0.5$ with an average settling velocity of 44 m/h and sludge volume index (SVI) of 34 mL/g. Total biomass concentration was 8.2 g/L, which was observed to be beneficial for the performance of the system. The extracellular polymeric substances (EPS) of newly developed biogranules were also measured in this study. The total EPS content for these biogranules was 0.083 g. SBR system containing MPAC biogranules showed the best removal performance when operated with 24 hours hydraulic retention time (HRT) with an intermittent of anaerobic (18 hours) and aerobic (6 hours) reactions. The highest removal performance for color, ammonia, TOC and COD were 83%, 98%, 95% and 97%, respectively. The final stage of the study involved the development of an artificial neural network (ANN) for the prediction of the biogranules performance at different HRT and reaction phases. The ANN model has successfully predicted the color removal performance with regression (R^2) of 0.9923 and mean square errors (MSE) of $2.75e^{-05}$. This study demonstrated that the addition of MPAC in the development of biogranules has demonstrated significant improvement in the physical, biological and chemical characteristics of the newly developed biogranules. The addition of MPAC could shorten and improve the biogranulation development where MPAC acts as the support media for microbial attachment during the development of biogranules.

ABSTRAK

Teknologi biogranulasi adalah suatu yang baru dalam bidang rawatan air sisa biologi dengan potensi penyingkiran yang tinggi serta mempunyai kelebihan dari segi ekonomi dan teknikal. Teknologi ini telah diuji secara meluas dalam merawat pelbagai jenis air sisa oleh kerana sifat enapcemar yang unik dan potensi biodegradasi yang tinggi. Walaupun cirinya unik, kekurangan utama biogranulasi adalah tempoh pembentukannya mengambil masa yang panjang. Kajian ini menyiasat kemungkinan untuk menghasilkan biogranul dengan serbuk karbon aktif magneti (MPAC) dalam merawat air sisa tekstil sintetik. Kajian ini bertujuan untuk meningkatkan proses pembentukan biogranul dengan ciri yang lebih baik dan prestasi penyingkiran yang tinggi. Pada peringkat awal kajian ini, kesan medan magnet dan MPAC terhadap pembentukan awal biogranul dikaji menggunakan satu faktor pada satu masa (OFAT) dan kaedah gerak balas permukaan (RSM). Pembentukan biogranul kemudiannya dikaji menggunakan dua reaktor kumpulan sesekumpul (SBR) berskala makmal dalam keadaan berselang seli bagi fasa anaerobik dan aerobik. Reaktor R1 bertindak sebagai sistem kawalan manakala Reaktor R2 ditambah dengan MPAC. Reaktor ini direka untuk beroperasi dengan jumlah isipadu 3 L dan dikendalikan dengan kadar sisa pertukaran isipadu (VER) 50%. Biogranul ini dihasilkan dengan menggunakan campuran enapcemar dari kilang tekstil dan sistem rawatan air sisa bandaran. Sistem ini dijalankan dengan air sisa tekstil sintetik. Prestasi penyingkiran, aspek struktur dan pembentukan biogranul MPAC telah dikaji berdasarkan sifat fizikal, biologi dan kimia. Keputusan kajian kelompok menunjukkan induksi medan magnet statik dan MPAC memberikan kesan positif yang signifikan untuk meningkatkan proses pembentukan biogranulasi di peringkat awal. Selepas 60 hari pembentukan, saiz purata biogranul meningkat dan mencapai $2.0 \text{ mm} \pm 0.5$ dengan halaju enapan 44 m/j dengan indeks isipadu enapcemar (SVI) 34 mL/g . Jumlah kepekatan biomas adalah 8.2 g/L , yang mana diperhatikan memberi kelebihan kepada prestasi sistem. Analisis kadar pengambilan oksigen (OUR) menunjukkan kehadiran bakteria fakultatif, anaerobik dan aerobik di dalam biogranul yang dihasilkan. Bahan polimer ekstraselular (EPS) daripada biogranul baru yang dihasilkan juga diukur dalam kajian ini. Jumlah kandungan EPS untuk biogranul ini adalah 0.083 g . Sistem SBR yang mengandungi biogranul MPAC menunjukkan prestasi penyingkiran yang baik apabila dikendalikan dengan masa tahanan hidraul (HRT) 24 jam dengan reaksi anaerobik (18 jam) dan reaksi aerobik (6 jam). Prestasi penyingkiran tertinggi untuk warna, ammonia, TOC dan COD masing-masing adalah 83%, 98%, 95% dan 97%. Tahap akhir kajian ini melibatkan pembentukan model *Artificial Neural Network* (ANN) untuk meramalkan prestasi biogranul di HRT dan fasa tindak balas yang berbeza. Model ANN telah berjaya meramalkan prestasi penyingkiran warna dengan regresi (R^2) 0.9923 dan ralat min kuasa dua (MSE) $2.75e^{-05}$. Kajian ini menunjukkan bahawa penambahan MPAC dalam pembentukan biogranul menunjukkan penambahbaikan yang signifikan dalam ciri fizikal, biologi dan kimia bagi biogranul yang baru dihasilkan. Penambahan MPAC dapat memendekkan masa dan meningkatkan pembentukan biogranulasi di mana MPAC bertindak sebagai media sokongan untuk pembiakan mikrob semasa pembentukan biogranul.

TABLE OF CONTENTS

	TITLE	PAGE
	DECLARATION	iii
	DEDICATION	iv
	ACKNOWLEDGEMENT	v
	ABSTRACT	vi
	ABSTRAK	vii
	TABLE OF CONTENTS	viii
	LIST OF TABLES	xiii
	LIST OF FIGURES	xvi
	LIST OF ABBREVIATIONS	xx
	LIST OF SYMBOLS	xxii
	LIST OF APPENDICES	xxiii
CHAPTER 1	INTRODUCTION	1
	1.1 Background of the Study	1
	1.2 Problem Statement	2
	1.3 Objectives of the Study	4
	1.4 Scope of the Study	4
	1.5 Significance of the Study	5
	1.6 Organization of Thesis	6
CHAPTER 2	LITERATURE REVIEW	7
	2.1 Introduction	7
	2.2 Biogranulation	7
	2.2.1 Microbial Immobilization	8
	2.2.3 Types of biogranules	9
	2.2.4 Development of Aerobic Granulation	13
	2.2.5 Factor Effecting Aerobic Granulation	14
	2.2.5.1 Substrate Composition	14

2.2.5.2	Organic Loading Rate	15
2.2.5.3	Reactor Configuration	17
2.2.5.4	Environmental Factor	19
2.2.6	Granule Characterization	22
2.2.6.1	Physical parameters	23
2.2.6.2	Chemical Parameters	28
2.2.6.3	Biological Parameters	29
2.3	Role of Extracellular Polymeric Substances in Biogranulation Development	31
2.3.1	Composition of Extracellular Polymeric Substances in Biogranules	32
2.3.2	Role of EPS on the Formation and Characteristic of Biogranules	33
2.3.3	Factor that Influent EPS Production in Biogranules Development	35
2.3.3.1	Substrate Type	35
2.3.3.2	Growth Phase	36
2.3.3.3	External Conditions	37
2.4	Static Magnetic Field	39
2.4.1	Magnetic Fields in Experimental Systems	39
2.4.2	Static Magnetic Field in Water and Wastewater Treatment	40
2.4.3	Effect of Static Magnetic Field on Biological Wastewater Treatment	42
2.5	Textile Wastewater	47
2.5.1	Characteristics of Textile Wastewater	49
2.6	Artificial Neural Network	52
2.6.1	Input and Output Variables	53
2.6.2	Transfer Function	54
2.6.3	Hidden Layer and Hidden Neuron	55

CHAPTER 3	RESEARCH METHODOLOGY	57
3.1	Introduction	57
3.2	Materials	58
3.2.1	Synthetic Wastewater	58
3.2.2	Seed Sludge	58
3.2.3	Chemical/Reagent and Equipment	51
3.2.4	Batch Experimental Set-Up	64
3.2.5	Reactor Set-up	67
3.4	Analytical Methods	69
3.4.1	Surface Hydrophobicity	70
3.4.2	Aggregation	70
3.4.3	Biomass Profile	71
3.4.4	Characterization of Biogranules	72
3.4.4.1	Settling Velocity	72
3.4.4.2	Sludge Volume Index	72
3.4.4.3	Granular Strength	73
3.4.5	Extracellular Polymeric Substances	74
3.4.5.1	Extraction by Heat Method	74
3.4.5.2	Determination of Carbohydrate Component	75
3.4.5.3	Determination of Polysaccharides Component	75
3.4.5.4	Determination of Protein Component	76
3.4.6	Field-emission Scanning Electron Microscope (FESEM)	77
3.4.7	Stereo Microscopic Examination	77
3.4.8	Determination of Dye Decolorization	78
3.4.9	Removal Performance Analysis	78
3.5	Experimental Procedure	79
3.5.1	Magnetic Activated Carbon	79
3.5.2	Batch Experiment	79

3.5.2.1	Effect of SMF on the Initial Biogranulation Process (Phase 1)	79
3.5.2.2	Effect of Biomass and MPAC on the Initial Biogranulation Process (Phase 2)	80
3.5.2.3	Experimental Design	80
3.5.3	Development and Post Development of Biogranulation	83
3.6	Artificial Neural Network	86
3.7	Data Analysis	88
CHAPTER 4	RESULT AND DISCUSSION	91
4.1	Introduction	91
4.2	Effect of Static Magnetic Field on the Initial Biogranulation	91
4.2.1	Effect of Static Magnetic Field on Surface hydrophobicity and Aggregation (Phase 1)	92
4.2.2	Effect of Static Magnetic Field on COD Removal	97
4.3	Characteristic of Magnetic Activated Carbon	98
4.4	Effect of MPAC and Biomass on Initial Biogranulation Process by Response Surface Methodology (Phase 2)	100
4.4.1	Factorial analysis: The Main Effect of MPAC and Biomass Concentration on Surface Hydrophobicity	101
4.4.2	Factorial analysis: The Interaction Effect of MPAC Biomass Concentration on Surface Hydrophobicity	105
4.4.3	Response Surface Analysis	107
4.5	Development of MPAC Biogranules	116
4.5.1	Biomass Concentration	116
4.5.2	Settling Velocity and Sludge Volume Index of the Biogranules	118
4.5.3	Size and Morphology of Biogranules	120
4.5.4	Cellular Characterization of Biogranules	125
4.5.5	Granular strength	128
4.5.6	Microbial Activity of the Biogranules	130

4.5.7	Total Extracellular Polymeric Substances	
	Content	132
4.5.7.1	Chemical Components of Extracellular Polymeric Substances	120
4.6	Removal Performance of Biogranules	138
4.6.1	Performance of Biogranules during Development Stage	138
4.6.2	Performance of Biogranules during Post-Development Stage	143
4.6.2.1	Effect of Hydraulic Retention Time on COD Removal	144
4.6.2.2	Effect of Hydraulic Retention Time on Color Removal	146
4.6.2.3	Effect of Hydraulic Retention Time on Ammonia Removal	147
4.6.2.4	Effect of Hydraulic Retention Time on TOC Removal	148
4.7	Prediction of Color Removal Performance using Artificial Neural Network	149
4.7.1	Prediction of Color Removal Performance at different HRT using Artificial Neuron Network	151
4.7.2	Prediction of Color Removal Performance with different duration phase using Artificial Neuron Network	155
CHAPTER 5	CONCLUSION AND RECOMMENDATIONS	161
5.1	Conclusions	161
5.2	Recommendations	162
REFERENCES		165

LIST OF TABLES

TABLE NO.	TITLE	PAGE
Table 2.1	Comparison of aerobic and anaerobic granulation process (Thanh et al, 2009)	12
Table 2.2	Support materials for biogranules development and their characteristic	21
Table 2.3	Summarize of factors effecting biogranules development	22
Table 2.4	Granulation treatment system with reaction phase, characteristic and removal performance of SBR system in treating variety types of wastewater	24
Table 2.5	Species dominancy in biogranules with different wastewater and sludge	30
Table 2.6	Effect of static magnetic field on various types of wastewater treatment	44
Table 2.7	Acceptable conditions for discharge of industrial effluent or mixed effluent of standards A and B	50
Table 2.8	Characteristics of textile wastewater	52
Table 3.1	Composition of synthetic textile wastewater	60
Table 3.2	Composition of trace element solution	60
Table 3.3	List of chemical/reagents used in the experiment	62
Table 3.4	List of equipment used in the experiment	63
Table 3.5	Compositions of Solution X, Solution Y and Solution Z for preparation of Lowry solution	77
Table 3.6	List of analytical method for removal performance analysis	78
Table 3.7	The variable and their limits value in the experiments	81
Table 3.8	2-level fractional factorial design in coded units	82
Table 3.9	CCD experimental runs in coded units	82
Table 3.10	One complete cycle of the SBR system for the development process	84
Table 3.11	One complete cycle of the SBR system for post development stage	85

Table 3.12	Dosage of carbon source for post-development period	86
Table 3.13	Experimental data for ANN model training	87
Table 4.1	Two-way ANOVA of surface hydrophobicity at different exposure time and magnetic field intensity	93
Table 4.2	Two-way ANOVA of aggregation process at different exposure time and magnetic field intensity	95
Table 4.3	Two-way ANOVA of initial turbidity between different exposure time and magnetic field intensity	96
Table 4.4	Two-way ANOVA of COD removals at different exposure time and magnetic field intensity	98
Table 4.5	Physical characteristics of MPAC	99
Table 4.6	Experimental result for 2-level factorial design analysis	100
Table 4.7	The p-value of the estimated main and interaction effect of sludge concentration and MAC on % SHb for 0-24 hours aeration time	101
Table 4.8	Experimental result for CCD analysis	108
Table 4.9	Summary of the p-value from the response surface modelling analysis	109
Table 4.10	ANOVA of mixed liquid suspended solid between reactor R1 and R2	118
Table 4.11	ANOVA of settling velocity between reactor R1 and R2	119
Table 4.12	ANOVA of sludge volume index between reactor R1 and R2	119
Table 4.13	ANOVA of integrity coefficient between reactor R1 and R2	130
Table 4.14	Concentration of EPS in matured biogranules for reactor R1 and R2	133
Table 4.15	ANOVA of COD removal between reactor R1 and R2	139
Table 4.16	ANOVA of color removal between reactor R1 and R2	141
Table 4.17	Wavelength and Absorbance for Peaks of UV-Vis for reactor R1 and reactor R2	143
Table 4.18	Input data testing of ANN model for color removal performance at different HRT	152
Table 4.19	ANN model performance analysis	154

Table 4.20	Input data testing of ANN model for colour removal performance with different duration phase	156
Table 4.21	ANN model performance analysis	159

LIST OF FIGURES

FIGURE NO.	TITLE	PAGE
Figure 2.1	Schematic diagram of aerobic granulation developed without any carrier material	11
Figure 2.2	Two forms of EPS	32
Figure 2.3	The main pollutants discharged from each step of textile wet processing	51
Figure 2.4	ANN structure consisting of input layer, hidden layer and output layer	54
Figure 2.5	Transfer function in ANN	55
Figure 3.1	Framework of the experimental study	59
Figure 3.2	(a) Indah Water Konsortium's (IWK) sewage treatment plant, Taman Impian Emas, Skudai and (b) Textile wastewater treatment plant Ramatex Textile Industrial Sdn. Bhd, Sri Gading Industrial Park, Batu Pahat.	61
Figure 3.3	Experimental set-up for Phase 1	65
Figure 3.4	Experimental set-up for Phase 2	66
Figure 3.5	Schematic layout of the SBR system	68
Figure 3.6	The SBR system used in this study	69
Figure 3.7	Graphical users Interface of MATHWorks MATLAB R2016a for neural network model	88
Figure 4.1	Effect of magnetic field on cell surface hydrophobicity in mixture of synthetic wastewater and biomass	93
Figure 4.2	Effect of magnetic field on aggregation process in mixture of synthetic wastewater and biomass	94
Figure 4.3	Effect of initial concentration of turbidity towards magnetic field in mixture of synthetic wastewater and biomass	95
Figure 4.4	Effect of magnetic field on COD removal performance in mixture of synthetic wastewater and biomass	98
Figure 4.5	Magnetic powder activated carbon	99

Figure 4.6	The pareto chart of the percentage surface hydrophobicity for (a) 0, (b) 3, (c) 6, and (d) 24 hours aeration time	102
Figure 4.7	Main effect plot for surface hydrophobicity for (a) 0 hours, (b) 3 hours, (c) 6 hour and (d) 24 hours	104
Figure 4.8	Interactive effect between MPAC and biomass concentration for surface hydrophobicity for (a) 0, (b) 3, (c) 6, and 24 hours aeration time (· Centre point)	106
Figure 4.9	Predicted versus actual data of surface hydrophobicity for (a) 0, (b) 3, (c) 6, and 24 hours aeration time	110
Figure 4.10	Contour (a) and 3D response surface (b) plots representing the relationship between variable (MPAC and biomass concentration) and surface hydrophobicity for 0 hours aeration time	112
Figure 4.11	Contour (a) and 3D response surface (b) plots representing the relationship between variable (biomass and MPAC concentration) and surface hydrophobicity for 3 hours aeration time	113
Figure 4.12	Contour (a) and 3D response surface (b) plots representing the relationship between variable (MPAC and biomass concentration) and surface hydrophobicity for 6 hours aeration time	114
Figure 4.13	Contour (a) and 3D response surface (b) plots representing the relationship between variable (MPAC and biomass concentration) and surface hydrophobicity for 24 hours aeration time	115
Figure 4.14	The profile of biomass concentration in SBR for reactor R1 and R2	117
Figure 4.15	The relationship between SVI value and SV for reactor R1 and R2	119
Figure 4.16	Microscopic image (a) Week 1, (b) Week 2, (c) Week 3, (d) Week 4, (e) Week 5, (f) Week 6, (g) Week 7, (h) Week 8 of microbial granules development for reactor R1 and R2 (Images were taken at magnification 6.7x, scale bar = 1 mm)	121
Figure 4.17	Microscopic image of matured MPAC biogranules R2 (Images were taken at magnification 30x, scale bar = 1 mm)	123
Figure 4.18	Flow of biogranulation process which involves of MPAC, EPS and filamentous bacteria	124
Figure 4.19	MPAC restrict filamentous bacteria from extending outside and shape the biogranules	124

Figure 4.20	FESEM microstructure observations on activated sludge under the magnification of (a) 10k (b) 25k	126
Figure 4.21	FESEM microstructure observations on mature biogranules for reactor R1 under the magnification of (a) 15k (b) 25k	126
Figure 4.22	FESEM microstructure observations on mature biogranules for reactor R2 under the magnification of (a) 15k (b) 25k	126
Figure 4.23	High power microscopic image of (a-b) activated sludge, (c) mature biogranules for reactor R1 and (d) mature biogranules for reactor R2 (Images were taken at magnification 400x, scale bar = 200 μ m)	127
Figure 4.24	MPAC cultivated with overgrown filamentous bacteria by acting as a framework to form large granules	128
Figure 4.25	The profile of integrity coefficient representing the granular strength of biogranules	129
Figure 4.26	The profile of dissolved oxygen in one complete cycle of the biogranules system for reactor R1 and R2	130
Figure 4.27	OUR profile in one complete cycle of aeration phase for reactor R1 and R2	131
Figure 4.28	BSA standard curves for proteins determination	134
Figure 4.29	Glucose standard curves for polysaccharides determination	134
Figure 4.30	Glucose standard curves for carbohydrates determination	135
Figure 4.31	Composition of chemical components in each LB-EPS and TB-EPS for reactors R1 and R2	135
Figure 4.32	The Laplace-Lorentz force on a moving charged and the magnetic field direction	137
Figure 4.33	The profile of COD removal for reactor R1 and R2	139
Figure 4.34	The profile of Color removal for reactor R1 and R2	140
Figure 4.35	UV- VIS spectrum for (a) reactors R1, (b) reactor R2 and (c) combination of both reactors	142
Figure 4.36	Profile of biomass concentration (MLSS) throughout the experiment during post-development stage for reactor R1 and R2	144
Figure 4.37	Profile of COD removal performance for both reactors R1 and R2 during post-development stage	145

Figure 4.38	Profile of color removal performance for both reactors R1 and R2 during post-development stage	146
Figure 4.39	Profile of ammonia removal performance for both reactors R1 and R2 during post-development stage	147
Figure 4.40	Profile of total organic carbon removal performance for both reactors R1 and R2 during post-development stage	148
Figure 4.41	ANN Model prediction of color removal performance for MPAC biogranules	150
Figure 4.42	Regression analysis of ANN model prediction for measured experimental data	150
Figure 4.43	ANN model prediction of color removal performance for MPAC biogranules at different HRT	153
Figure 4.44	Regression analysis of ANN model prediction at HRT of 12 hours	153
Figure 4.45	Regression analysis of ANN model prediction at HRT of 24 hours	154
Figure 4.46	Regression analysis of ANN model prediction at HRT of 36 hours	154
Figure 4.47	ANN model prediction of colour removal performance for MPAC biogranules with different duration phase	157
Figure 4.48	Regression analysis of ANN model prediction for type 1	157
Figure 4.49	Regression analysis of ANN model prediction for type 2	158
Figure 4.50	Regression analysis of ANN model prediction for type 3	158

LIST OF ABBREVIATIONS

16S rRNA	-	16 subunit ribosomal ribonucleic acid
ADMI	-	American Dye Manufacturing Index
Ag	-	Aggregation (%)
ANN	-	Artificial Neural Network
ANOVA	-	Analysis of variance
APHA	-	American Public Health Association
BSA	-	Bovine serum albumin
CCD	-	Central Composite Design
COD	-	Chemical oxygen demand (mg/L)
DGGE	-	Denaturing gradient gel electrophoresis
DO	-	Dissolved oxygen (mg/L)
EPS	-	Extracellular polymeric substances
FISH	-	Fluorescent <i>in situ</i> hybridization
FESEM	-	Field-Emission Scanning Electron Microscope
HRT	-	Hydraulic retention time (h)
IC	-	Integrity coefficient (%)
IPC	-	Integrated pollution control
IWK	-	Indah Water Konsortium
LOFT	-	Lack of fit test
LB-EPS	-	Loosely-bound Extra-cellular polymeric substances
MPAC	-	Magnetic powder activated carbon
MSE	-	Mean square error
MLSS	-	Mixed liquor suspended solid (mg/L)
MLVSS	-	Mixed liquor volatile suspended solid (mg/L)
MWTP	-	Municipal wastewater treatment plan
OLR	-	Organic loading rate ($\text{kg}/\text{m}^3 \cdot \text{day}$)
OUR	-	Oxygen uptake rate ($\text{mg}/\text{L} \cdot \text{h}$)
PN	-	Exoprotein
PS	-	Polysaccharide

RG	-	Residual granules (mg)
RSM	-	Response surface method
SAV	-	Superficial air velocity (cm/s)
SBR	-	Sequencing batch reactor
SMF	-	Static Magnetic Field
SG	-	Settled granules (mg)
SV	-	Settling velocity (cm/s)
SHb	-	Surface hydrophobicity
SRT	-	Sludge retention time (day)
SVI	-	Sludge volume index (mL/g)
STP	-	Sewage treatment plant
SS	-	Suspended solid
SOUR	-	Specific oxygen uptake rate
TB-EPS	-	Tightly-bound Extra-cellular polymeric substances
TOC	-	Total organic carbon (mg/L)
UV-Vis	-	Ultraviolet visible spectroscopy
VER	-	Volumetric exchange rate
WW	-	Wastewater
WWTP	-	Wastewater treatment plant

LIST OF SYMBOLS

$^{\circ}\text{C}$	-	Celsius
R^2	-	Coefficient of determination
T_i	-	Turbidity influent (initial)
T_f	-	Turbidity effluent (final)

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
Appendix A	DATA AND CALCULATIONS FOR EXPERIMENTAL ANALYSIS (PHASE 1)	191
Appendix B	FACTORIAL DESIGN AND RESPONSE SURFACE METHODOLOGY DATA ANALYSIS FOR SURFACE HDROPHOBICITY (PHASE 2)	196
Appendix C	DATA AND EXAMPLES OF CALCULATIONS FOR DEVELOPMENT OF BIOGRANULES	202
Appendix D	DATA AND EXAMPLES OF CALCULATIONS FOR POST- DEVELOPMENT OF BIOGRANULES	208

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

The rapid population growth and industrialization has caused an increase on the volume of wastewater disposed into the environment. Industrialization in the textile industry is synonymous with the consumption of large volume of water which is subsequently disposed as wastewater containing high load pollutants. These pollutants can cause contamination to water body if there are not properly eliminated from wastewater.

Several treatment technologies can be utilized for textile wastewater treatment. In Malaysia, most industrial effluents are treated with conventional wastewater treatment processes involving physical, chemical and biological techniques. Most treatment plants use single or combined biological treatment process comprising of aerobic, anoxic and anaerobic systems. Application of physical and chemical processes in these treatment plants are hindered by their associated capital and operational cost (Holkar et al, 2016). Furthermore, excessive application of chemical during treatment can cause secondary pollution. Some of the treatment systems can potentially remove color from wastewater while most treatment processes transform contaminants into different forms. According to Integrated Pollution Control (IPC) regulation, decoloration systems that transfer pollutants between environments are prohibited (Willmott et al, 1998).

Biogranulation technology is one of the great achievements in biological wastewater treatment of the twentieth century. It is a compact and dense microbial aggregate formed through self-microbial immobilization involving physical, chemical and biological processes (Liu and Tay, 2004). Biogranules are

differentiated from conventional activated sludge systems by their regular shape, dense nature, strong microbial structure and good settleability (Zheng et al, 2006). This biogranules consist millions of microorganism that clump together with anaerobic microorganism occupying the inner layer and aerobic microbe at the outer layer of the biogranules. The presence of both types of microorganisms in the granules makes biogranulation a suitable technology for the complete biodegradation of textile wastewater.

Biogranules are usually developed using sequencing batch reactors (SBR) with cycle configuration strictly regulated for rapid settling and frequent repetition of feast and famine condition. This configuration supports the growth of dense and stable biogranules. However, studies have shown that properties of biogranules developed in SBR are affected to several factors including organic loading rate, substrate composition, feast-famine regime, hydrodynamic shear force, feeding strategy, reactor configuration, dissolved oxygen (DO), cycle time, volume exchange ratio, solids retention time and settling time.

1.2 Problem Statement

Biogranules application in wastewater treatment is considered as promising alternative in biotechnology. Biogranulation is associated with several advantages such as high settling velocity and strong microbial structure which causes high sludge retention and tolerate higher loading rates from high strength wastewater. The characteristic of biogranules that have various types of microorganisms would able to perform both aerobic and anaerobic degradation process in a single reactor column makes biogranulation technology suitable for degradation of textile wastewater. Textile wastewater is known as a complex chemical structure and studies have shown that complete mineralization of dye compound in textile wastewater required both anaerobic and aerobic biological approaches (Melgoza et al, 2004). Hence, biogranulation systems seem to be a suitable biological treatment approach that may be able to perform a complete and effective degradation process for textile wastewater.

However, the long start-up period and instability of the reactor system under long SBR operation has been the major drawback of granulation technology. In order to enhance fast biogranulation process and as well as to enhance the stability of biogranules, many attempts have been conducted by adding various types of substances during development of biogranules such as granular activated carbon (Li et al, 2012; Zhou et al, 2015; Tao et al, 2017), zeolite (Wei et al, 2012), dry sewage sludge micropowder (Li et al, 2015), yellow earth (He et al, 2016) and magnetic nanoparticles (Liang et al, 2017). The use of these materials increases the aggregation percentage of microorganisms by acting as nuclei during biogranules development (Li et al, 2015). Previous research reported that static magnetic field able to enhance biogranulation development (Wang et al, 2012; Liu et al, 2016). Then several attempts have been made to enhance growth of microbes using magnetic field induction (Nakamura et al, 1997; Motta et al, 2001; Muniz et al, 2007; Novak et al, 2007, Tu et al, 2015) and increase EPS production (Wang et al, 2012).

However, the effect of SMF on the start-up period of the bioreactor, aggregation, hydrophobicity, settleability and flocculation ability of microbial granules was not much reported. The application of specific carriers acted together with magnetized sludge on the biogranules development process is very much lacking. In particular, there is lack of information relating to the effect of SMF and specific carriers on EPS production and biogranules development. Thus, a comprehensive study on the application of specific carriers and SMF in the development of biogranules is needed.

This study proposed a different approach to accelerating the biogranulation process. The magnetic powder activated carbon (MPAC) was evaluated as a potential enhancer of biogranulation development process. The system utilized the concept of sequential anaerobic and aerobic biological reactions for complete degradation of textile wastewater. Furthermore, magnetic field concept was used to initiate and enhance the initial granulation stage. The newly formed biogranules were characterized and their performance evaluated for its performance in order to observe the impact of the addition of magnetic powder activated carbon onto biogranules.

1.3 Objectives of the Study

The objective of this study are:

- i. To investigate the effect of static magnetic field and magnetic powder activated carbon at the initial stage of biogranulation development through batch study.
- ii. To develop biogranules with the addition of magnetic powder activated carbon and characterizes the newly developed biogranules for its physical, chemical and biological properties.
- iii. To investigate the removal performance of the enhanced biogranulation system during the development and post-development stage.
- iv. To develop an artificial neural network (ANN) model for prediction of color removal performance of MPAC biogranules.

1.4 Scope of the Study

This study covers the design and application of batch test experiment and a laboratory-scale reactor system that are based on the sequential batch reactor system. All of the experiments were conducted in Environmental Laboratory, School of Civil Engineering, Universiti Teknologi Malaysia (UTM).

Initially batch experiments were carried out to investigate the effect of magnetic field intensity and MPAC concentration on the aggregation and surface hydrophobicity. Then, biogranules were developed using synthetic textile wastewater with combination of MPAC. The cultivation of biogranules was investigated using two laboratory scale SBR under intermittent anaerobic and aerobic conditions. The SBR were operated in parallel with Reactor R1 as control and Reactor R2 containing

of MPAC. During development process, samples of biogranules were collected and examined. The physical, chemical and biological properties of matured biogranules were characterized. The reactor performances for post-development were studied based on COD, TOC, ammonia and color removal. Dye degradation in the treated wastewater was measured using ultraviolet visible spectroscopy (UV-Vis). Furthermore, field-emission scanning electron microscope analysis (FESEM) was used to inspect the microstructural characteristics of matured biogranules. The study also included analysis of extracellular polymeric substances (EPS) of biogranules. EPS compositions consist of proteins, polysaccharides and carbohydrates were determined in this study. Finally, an artificial neural network (ANN) model was developed to predict the performance of MPAC biogranules in term of color removal.

1.5 Significance of the Study

Biogranulation technology is a promising method for wastewater treatment due to its low operational and investment cost as well as small space requirement (Liu et al, 2010). Recently, various approaches have been used by to improve the startup period of biogranules. Improving the initial biogranulation development stage could enhance the efficiency of biogranules. However, the influence of magnetic field combined with magnetic activated carbon is not yet evaluated. There is a lack of information on the effect of magnetic field and magnetic activated carbon on biogranules development. Therefore, the significance of this study can be listed as follows.

- i. The study investigates the effect of magnetic field (intensity 0-30 mT) on aggregation, surface hydrophobicity and COD performance of activated sludge. It further examines the impact of magnetic activated carbon on activated sludge for initial biogranules development. The findings would provide knowledge on suitable conditions for the development of the MPAC biogranules

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