

**DYNAMIC ADSORPTION AND REGENERATION STUDIES OF
ACID YELLOW 17 USING MODIFIED ACTIVATED CARBON**

LAVANYHA A/P GURU BALAN

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**DYNAMIC ADSORPTION AND REGENERATION STUDIES OF
ACID YELLOW 17 USING MODIFIED ACTIVATED CARBON**

**by
LAVANYHA A/P GURU BALAN**

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TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENT	ii
TABLE OF CONTENTS	iii
LIST OF TABLES	viii
LIST OF FIGURES	x
LIST OF SYMBOL	xiv
LIST OF ABBREVIATIONS	xvi
ABSTRAK	xvii
ABSTRACT	xviii
CHAPTER ONE: INTRODUCTION	1
1.1 Research Background	1
1.1.1 Dyes Pollution	1
1.1.2 Activated Carbons	3
1.1.3 Fixed Bed Column Adsorption Study	4
1.2 Problem Statement	5
1.3 Research Objectives	6
1.4 Research Scope	6
1.5 Organization of Thesis	7
CHAPTER TWO: LITERATURE REVIEW	8

2.1	Dyes	8
2.1.1	Classification Systems for Dyes	8
2.1.2	Reactive Dyes	11
2.1.3	Technologies For Dye Removal	11
2.1.3 (a)	Biological Treatments	13
2.1.3 (b)	Chemical Methods	13
2.1.3 (c)	Physical Methods	14
2.2	Adsorbents	14
2.2.1	Activated Carbon	14
2.3	Adsorption	16
2.4	Adsorbents Used for Acid Dye Adsorption	17
2.4.1	Coconut Shell Activated Carbon	17
2.4.2	Metal Impregnated Activated Carbon	18
2.5	Continuous Fixed Bed Adsorption Column	19
2.5.1	Adsorption capacity analysis	19
2.5.2	Column Dynamic Adsorption Model	19
2.5.2 (a)	Thomas Model	20
2.5.2 (b)	Yoon-Nelson Model	21
2.5.2 (c)	Adams-Bohart Model	21

2.5.2 (d) Bed Depth/ Service Time analysis (BDST) Model	22
2.5.3 Error Analysis	22
2.6 Adsorbent reusability and regeneration	23
CHAPTER THREE: MATERIALS AND METHOD	25
3.1 Materials and Chemicals Required	25
3.2 Equipment and Facilities Required	26
3.3 Preparation of Activated Carbon	27
3.3.1 Coconut Shell Activated Carbon	27
3.3.2 Chromium Impregnated Coconut Shell Carbon	27
3.4 Adsorbate	28
3.4.1 Acid Yellow 17	28
3.4.2 Preparation of Dye Stock Solution	29
3.5 Analytical Technique	29
3.5.1 Double beam UV-VIS Spectrophotometer (UV-1800)	29
3.5.1 (a) Preparation of Calibration Curve	29
3.5.1 (b) Determination of concentration from absorbance	30
3.5.2 Atomic Absorption Spectrophotometer (AAS-665)	30
3.5.2 (a) Preparation of Calibration Curve	31
3.5.2 (b) Determination of concentration from absorbance	31

3.6	Determination of Optimal Impregnation Ratio	32
3.7	pH Screening	32
3.8	Determination of Point of Zero Charge of Chromium Impregnated Activated Carbon	33
3.9	Characterization of Adsorbent	33
	3.9.1 Brunauer-Emmett-Teller (BET) Analysis	33
	3.9.2 Fourier Transform Infrared (FTIR) Spectroscopy analysis	34
3.10	Experimental Procedure for Column Adsorption Studies	35
	3.10.1 Continuous Fixed Bed Adsorption Studies Set-up	35
	3.10.1 (a) Effect of initial dye concentration	37
	3.10.1 (b) Effect of inlet flow rate of dye solution	38
	3.10.1 (c) Effect of bed height of adsorbent	38
	3.10.2 Column Bed Regeneration	38
	CHAPTER 4: RESULTS AND DISCUSSION	40
4.1	Characterization of Adsorbents	40
	4.1.1 Brunauer-Emmett-Teller (BET) Analysis	40
	4.1.2 Fourier Transform Infrared (FTIR) Spectroscopy Analysis	43
	4.1.3 Point of Zero Charge	45
4.2	Fixed Bed Adsorption Study	46
	4.2.1 Effect of initial dye concentration	46

4.2.2	Effect of inlet flow rate of dye solution	48
4.2.3	Effect of bed height of adsorbent	50
4.2.4	Adsorption capacity analysis	52
4.3	Column Dynamics Study	53
4.3.1	Application of Thomas Model	53
4.3.2	Application of Yoon-Nelson Model	57
4.3.3	Application of Adams-Bohart Model	61
4.3.4	Application of Bed Depth/ Service Time analysis (BDST) Model	65
4.4	In situ Column Regeneration Study	66
	CHAPTER 5: CONCLUSION AND RECOMMENDATIONS	69
5.1	Conclusion	69
5.2	Recommendations	70
	REFERENCES	71
	APPENDICES	80
	APPENDIX A: UV-VIS SPECTROPHOTOMETER CALIBRATION CURVE	80
	APPENDIX B: AAS CALIBRATION CURVE FOR CHROMIUM	80

LIST OF TABLES

		Page
Table 2.1	Usage classification of dyes (Hunger, 2007).	9
Table 2.2	Principal existing and emerging processes for dyes removal (Crini, 2006).	12
Table 2.3	Summary of method for AC regeneration and reference for previous works.	24
Table 2.4	Optimal concentration of various solvents to desorb the yellow dyes from the activated carbon (Lu et al., 2011).	24
Table 3.1	List of materials and chemicals.	25
Table 3.2	List of equipment and facilities.	26
Table 3.3	Characteristics of Acid Yellow 17.	28
Table 3.4	List of items used in the continuous fixed bed adsorption.	36
Table 4.1	Summary of textual characteristics of modified AC.	41
Table 4.2	Summary of functional groups on the activated carbon and their corresponding infrared assignments.	45
Table 4.3	Dynamic adsorption capacity of AY 17 on CMAC at different experimental conditions.	52
Table 4.4	Thomas kinetic model parameters at different experimental conditions.	55
Table 4.5	Yoon-Nelson kinetic model parameters at different experimental conditions.	60

Table 4.6	Adams-Bohart kinetic model parameters at different experimental conditions.	64
Table 4.7	BDST parameters at 10 mL/min, 50 mg/L AY 17 adsorption onto CMAC adsorbent.	66
Table 4.8	Summary of the in situ column regeneration study.	68

LIST OF FIGURES

	Page
Figure 1.1 Contribution of textile industry to water pollution for different states in Malaysia (Pang and Abdullah, 2013).	2
Figure 2.1 Adsorption operations with solid particle adsorbent (Seader and Henley, 1998).	17
Figure 3.1 Structure of Acid Yellow 17.	28
Figure 3.2 Double beam UV-VIS Spectrophotometer (UV-1800).	29
Figure 3.3 Atomic Absorption Spectrophotometer (AAS-665).	31
Figure 3.4 Surface area and porosity analyser (ASAP 2020).	34
Figure 3.5 Fourier Transform Infrared Spectrophotometer (IRPrestige-21).	35
Figure 3.6 Continuous fixed bed adsorption study experimental set-up.	37
Figure 4.1 Nitrogen sorption isotherm plot for CMAC.	42
Figure 4.2 Differential pore volume distribution over the pore diameter.	43
Figure 4.3 FTIR spectroscopy of modified AC before and after AY 17 adsorption.	44
Figure 4.4 Point of zero charge (pH_{pzc}) of CMAC.	45
Figure 4.5 Experimental breakthrough curves for adsorption of AY 17 on CMAC at different initial dye concentration (conditions: inlet flow rate = 10 mL/min, bed height = 4 cm, temperature = 25 ± 1 °C) for the first 60 minutes.	47

Figure 4.6	Experimental breakthrough curves for adsorption of AY 17 on CMAC at different initial dye concentration for the first 30 minutes.	48
Figure 4.7	Experimental breakthrough curves for adsorption of AY 17 on CMAC at different inlet flow rate of dye solution (conditions: initial dye concentration = 50 mg/L, bed height = 4 cm, temperature = 25 ± 1 °C) for the first 60 minutes.	49
Figure 4.8	Experimental breakthrough curves for adsorption of AY 17 on CMAC at different inlet flow rate of dye solution.	50
Figure 4.9	Experimental breakthrough curves for adsorption of AY 17 on CMAC at different bed height of adsorbent (conditions: initial dye concentration = 50 mg/L, inlet flow rate = 10 mL/min, temperature = 25 ± 1 °C).	51
Figure 4.10	Experimental breakthrough curves for adsorption of AY 17 on CMAC at different bed height of adsorbent for the first 60 minutes.	51
Figure 4.11	(a) Thomas kinetic plot for the adsorption of AY 17 on CMAC for the first 60 min: effect of initial dye concentration (inlet flow rate = 10 mL/min, bed height = 4 cm, temperature = 25 ± 1 °C). (b) Thomas kinetic plot for the adsorption of AY 17 on CMAC for the first 60 min: effect of inlet flow rate of dye solution (initial dye concentration = 50 mg/L, bed height = 4 cm, temperature = 25 ± 1 °C). (c) Thomas kinetic plot for the adsorption of AY 17 on CMAC for the first 60 min: effect of	53

bed height (initial dye concentration = 50 mg/L, inlet flow rate = 10 mL/min, temperature = 25 ± 1 °C).

Figure 4.12 (a) Yoon-Nelson kinetic plot for the adsorption of AY 17 on CMAC for the first 60 min: effect of initial dye concentration (inlet flow rate = 10 mL/min, bed height = 4 cm, temperature = 25 ± 1 °C). (b) Yoon-Nelson kinetic plot for the adsorption of AY 17 on CMAC for the first 60 min: effect of inlet flow rate of dye solution (initial dye concentration = 50 mg/L, bed height = 4 cm, temperature = 25 ± 1 °C). (c) Yoon-Nelson kinetic plot for the adsorption of AY 17 on CMAC for the first 60 min: effect of bed height (initial dye concentration = 50 mg/L, inlet flow rate = 10 mL/min, temperature = 25 ± 1 °C).

Figure 4.13 (a) Adams-Bohart kinetic plot for the adsorption of AY 17 on CMAC: effect of initial dye concentration (inlet flow rate = 10 mL/min, bed height = 4 cm, temperature = 25 ± 1 °C). (b) Adams-Bohart kinetic plot for the adsorption of AY 17 on CMAC: effect of inlet flow rate of dye solution (initial dye concentration = 50 mg/L, bed height = 4 cm, temperature = 25 ± 1 °C). (c) Adams-Bohart kinetic plot for the adsorption of AY 17 on CMAC: effect of bed height (initial dye concentration = 50 mg/L, inlet flow rate = 10 mL/min, temperature = 25 ± 1 °C).

Figure 4.14 BDST plot for AY 17 at different values of C_t/C_0 (0.1 and 0.5).

Figure 4.15	Breakthrough curves for regeneration cycle AY 17 from CMAC.	67
Figure 4.16	Breakthrough curves for regeneration cycle AY 17 from CMAC for the first 35 minutes.	67
Figure A.1	UV-VIS spectrophotometer calibration curve.	80
Figure B.1	AAS calibration curve.	80

LIST OF SYMBOLS

Symbol		Unit
C_{ad}	Adsorbed AY 17 Concentration	$mg L^{-1}$
C_0	Inlet/Initial Dye Concentration	$mg L^{-1}$
C_t	Effluent Dye Concentration	$mg L^{-1}$
K_{BD}	BDST Rate Constant	$mL min^{-1} mg^{-1}$
K_{Th}	Thomas Rate Constant	$mL min^{-1} mg^{-1}$
K_{YN}	Yoon-Nelson Rate Constant	min^{-1}
K_{AB}	Adams-Bohart Rate Constant	$mL min^{-1} mg^{-1}$
N_{BD}	BDST Adsorption Capacity	$g L^{-1}$
N_o	Adsorption Capacity dependent of flow rate	$g L^{-1}$
$q_{e,av}$	Average Equilibrium Adsorbate Uptake	$mg g^{-1}$
$q_{e,cal}$	Calculated Equilibrium Adsorbate Uptake	$mg g^{-1}$
$q_{e,exp}$	Experimental Equilibrium Adsorbate Uptake	$mg g^{-1}$
q_i	i th Regenerated Column Capacity	$mg g^{-1}$
q_o	Fresh Column Capacity	$mg g^{-1}$
$q_{o,Th}$	Thomas Model Equilibrium Adsorbate Uptake	$mg g^{-1}$
$q_{o,YN}$	Yoon-Nelson Equilibrium Adsorbate Uptake	$mg g^{-1}$
q_{total}	Total Equilibrium Adsorbate Uptake	$mg g^{-1}$
Q	Flow Rate	$mL min^{-1}$
r^2	Square Of The Correlation Coefficient	-

t	Time	min
τ	Time Required For 50 % Adsorbate Breakthrough	min
U	Influent Linear Velocity	cm min ⁻¹
W_{total}	Total amount of AY 17 sent to the column	mg
x	Mass of Adsorbent	g
Z	Bed Depth in the Column	cm

LIST OF ABBREVIATIONS

AAS	Atomic Absorption Spectrophotometer
AB	Adams-Bohart
AC	Activated Carbon
AY 17	Acid Yellow 17
BET	Brunauer-Emmett-Teller
BJH	Barrett, Joyner, and Halenda
C.I	Colour Index
CMAC	Chromium Modified Activated Carbon
DE	Desorption efficiency
FTIR	Fourier Transform Infrared
IUPAC	International Union of Pure and Applied Chemistry
pH _{zpc}	Point of zero charge
SSE	Sum of squares of error
YN	Yoon-Nelson

**KAJIAN PENJERAPAN DINAMIK DAN PENJANAAN SEMULA PEWARNA
ASID KUNING 17 MENGGUNAKAN BUTIRAN KARBON TERAKTIF
DIUBAHSUAI**

ABSTRAK

Pencemaran air menjadi kebimbangan utama terutamanya pencemaran air oleh bahan pencelup hasil buangan industri tekstil. Untuk mengurangkan pencemaran yang disebabkan oleh bahan pencelup, kajian ini dijalankan untuk menentukan penggunaan butiran karbon teraktif yg diubahsuai dengan kromium (CMAC) untuk penjerapan pewarna Asid Kuning 17 (AY 17). Kajian penjerapan turus lapisan tetap dijalankan untuk mengkaji dan menentukan keadaan optimum untuk penjerapan AY 17 untuk berlaku. Untuk kajian penjerapan turus lapisan tetap, parameter yang disiasati ialah kesan kepekatan awal pewarna (10-50 mg/L), kadar kelajuan aliran (10-30 mL/min) dan ketinggian lapisan karbon (2-4 cm). Untuk kesan kepekatan awal pewarna, masa bulus adalah terpantas untuk 50 dan 40 mg/L. Untuk kesan kadar kelajuan aliran, lengkung bulus menjadi lebih curam apabila kadar kelajuan aliran meningkat. Untuk kesan ketinggian lapisan karbon, masa bulus meningkat apabila ketinggian lapisan karbon meningkat. Dinamik turus juga dikaji dan model yang digunakan adalah Thomas, Yoon-Nelson, Adams-Bohart dan model analisis BDST. Model Thomas, Yoon-Nelson, dan BDST amat serasi dengan data penjerapan berbanding model Adams-Bohart. Untuk kajian penjanaaan semula, masa bulus menurun dan lengkung bulus menjadi lebih curam apabila CMAC menjalani penjanaaan semula. Kapasiti penyerapan juga berkurang apabila kitaran penjanaaan semula dijalankan beberapa kali.

**DYNAMIC ADSORPTION AND REGENERATION STUDIES OF ACID
YELLOW 17 USING MODIFIED ACTIVATED CARBON**

ABSTRACT

Water pollution become an integral concern especially dye pollution which is waste generated by textile industry. In order to reduce the pollution caused by dyes, this study was conducted to determine the usage of chromium modified activated carbon (CMAC) for the adsorption of Acid Yellow 17 (AY 17) dye. Fixed bed adsorption study was conducted to investigate the optimum condition for adsorption of Acid Yellow 17 to occur. For the fixed bed adsorption study, the parameter that was studied was the effect of initial dye concentration (10-50 mg/L), inlet flow rate of AY 17 solution (10-30 mL/min) and bed height of adsorbent (2-4 cm). For the effect of initial dye concentration, the breakthrough time was the fastest for 50 mg/L and 40 mg/L. For the effect of inlet flow rate of AY17 solution, the breakthrough curve becomes steeper as the inlet flow rate of dye solution increases. For the effect of bed height of adsorbent, the breakthrough time increases as the bed height increases. Column dynamics was also studied and the model used was Thomas, Yoon-Nelson, Adams-Bohart, and BDST analysis model. Thomas, Yoon-Nelson and BDST analysis model fitted well the adsorption data as compared to Adams-Bohart model. For the in situ column regeneration study, the breakthrough time decreases and the slope of the curve becomes steeper as the CMAC undergoes series of regeneration. The adsorption capacity also decreases as the regeneration cycle is repeated a few times.

CHAPTER ONE

INTRODUCTION

1.1 Research Background

1.1.1 Dyes Pollution

Environmental protection has become an integral concern among citizens of the world as the expeditious growth of technologies threatens to harm the environment. Industrial practices and products generate wastes that are harmful to public health and the environment if these wastes are not properly treated. (Foo and Hameed, 2010). In Malaysia, the industrial development had contributed substantial benefits to the overall economic development. However, this had left significant effects on the environmental resources since all industries are required to utilize both renewable and non-renewable resources from the environment. This results in residues being discharged into the water as wastes due to the conversion of these resources into finished or semi- finished products. These wastes which are in the form of solid, liquid and gas affects the quality of the water (Muyibi et al., 2008).

In Malaysia, water pollution is mainly caused by point and non-point sources. Major point sources are sewage treatment plants, manufacturing, agro-based industries and animal farms while non-point sources include agricultural activities and surface runoffs. In 2009, textile industry contributed only about 0.1 % of the industrial sources of scheduled waste (solid waste) produced in Malaysia. Even though so, there was a sharp increase in the total amount of scheduled waste generated by textile industry which is from 744 tons in 2007 to 1559 tons in 2009. Besides that, 22 % of the total volume of industrial wastewater generated in Malaysia is accounted by textile finishing wastewater (Yeoh et al., 1993).

Figure 1.1 shows the contribution of textile industry to water pollution for different states in Malaysia (Pang and Abdullah, 2013). It can be observed that Peninsular Malaysia has the major sources of textile industry wastewater pollution as compared to West Malaysia. Johor state has the highest number of water pollution sources with 28.6 %, followed by Pulau Pinang (28.2 %) and Selangor (15.6 %) (Muyibi et al., 2008). The statistic is consistent with the number of textile finishing plants which are located at Johor, Pulau Pinang and Johor (Yeoh et al., 1993).

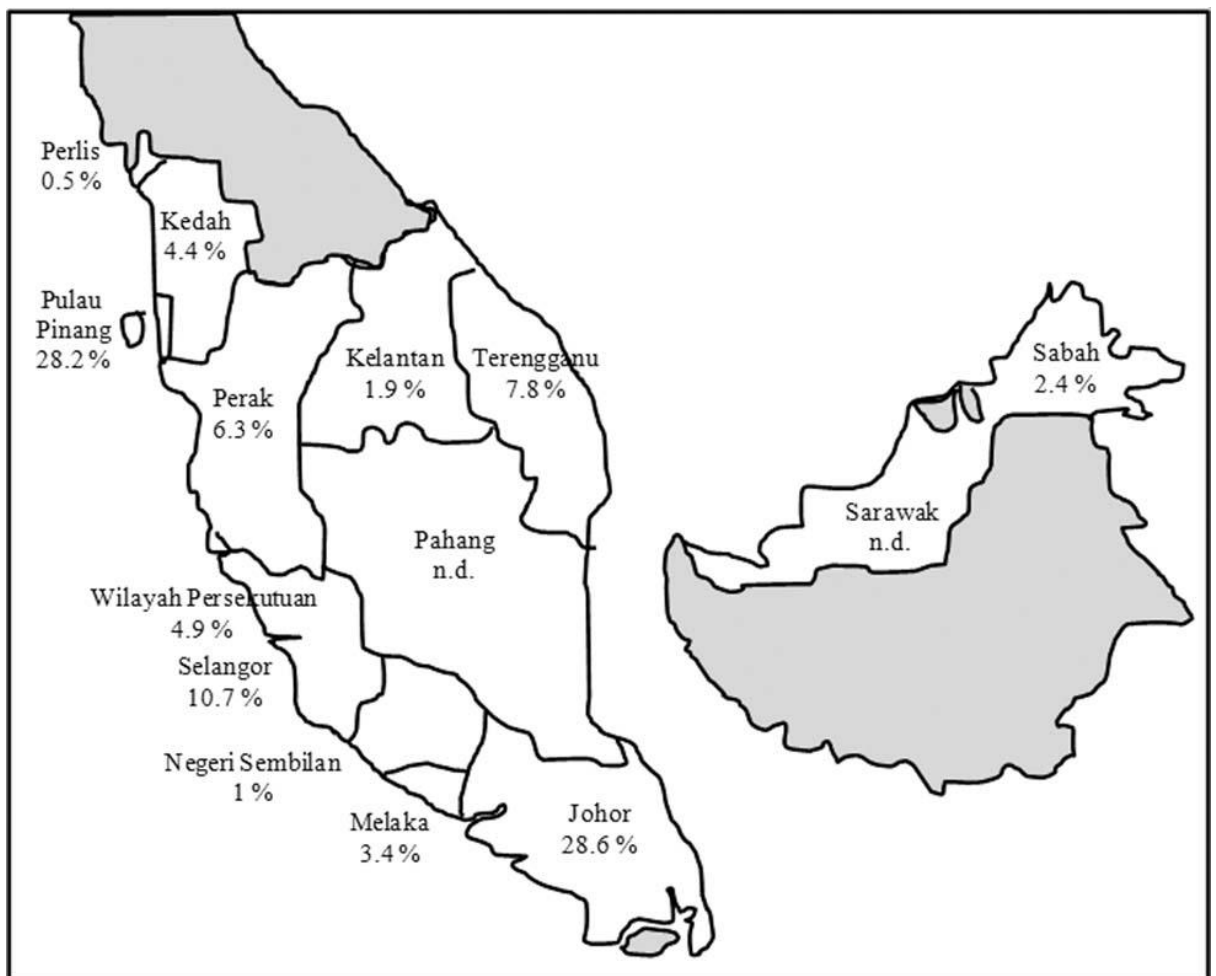


Figure 1.1 : Contribution of textile industry to water pollution for different states in Malaysia (Pang and Abdullah, 2013).

As reported by Gupta and Suhas (2009), there are more than 100000 commercial dyes overall at the present and the rough estimation for production is $7 \times 10^5 - 1 \times 10^6$ (Christie, 2007, Hunger, 2007, Husain, 2006). The exact data on the quantity of dyes that

is discharged to the environment of such a huge production is unfortunately unavailable. However, about 10-15% of used dyes enter the environment through wastes from various industries like textile, dyeing, paper and pulp, tannery and paint (Husain, 2006). With the ever increasing demand for textile products, it comes to no surprise that textile industries are rated as the main industry which is responsible for endless pollution of the environment among all the other industrial sectors in the world (Foo and Hameed, 2010).

If these textile effluent is not properly treated, the presence of azo- and nitro-compounds in the organic dyes structure could possibly generate aromatic amines which is likely to induce cancer and tumours in human. In addition, the presences of dyes as contaminants in wastewater can cause toxicological effects to aquatic life and human health. Metals can also be present in different dyes as textile colourants. These metals may pose negative impacts and hazard to human health as well as the environment. These hazards may include effects of consumption such as carcinogenic, genotoxic, mutagenic and teratogenic properties. Besides, presence of non-biodegradable nature and high colour intensity of organic dyes may block the passage of sunlight through water which can lead to the reduction of aquatic diversity. Some of these dyes, if swallowed, can cause irritations to the skin, eyes, respiratory tracts and allergic dermatitis (Pang and Abdullah, 2013).

1.1.2 Activated Carbons

To treat dye polluted water, adsorption is considered to be an attractive method (Ferreira et al., 2017). Adsorption seems to be proven suitable in removing organic pollutants due to its effectiveness and for economically reasonable (Garg et al., 2003). Activated carbon (AC) has generally been used as an adsorbent for the removal of dyes from aqueous solutions (Ferreira et al., 2017). AC has been extensively practiced in

numerous industrial processes including odour removal, gas separation, medicinal use, pharmaceutical use and in wastewater treatment due to AC having particularly large specific surface area, high pore volume, well-developed internal porous structure, high thermal stability, relative chemical stability and plenty of surface functional groups (Tran et al., 2017, Saygılı et al., 2015).

Activated carbons' adsorption performance is greatly affected by the porosity and surface chemical properties (Li et al., 2002, Wang et al., 2018). So, activated carbons, which are mainly synthesized through physical or chemical activation, undergo surface modifications to improve the adsorption capacity and selectivity of ACs (Tran et al., 2017, Lian et al., 2016). Therefore, surface modifications of ACs was recognized as a practical and effective method and currently surface modifications has gained huge attentions (Ahmed et al., 2016).

1.1.3 Fixed Bed Column Adsorption Study

In the past years, numerous studies of adsorption can be found and are reported in literatures. However, for large-scale wastewater treatment, it was determined that fixed bed columns are often the preferred method (Aksu and Çağatay, 2006). Fixed bed column adsorption proved to be advantageous due to its simple mode of operation and it can achieve a high removal efficiency. It can also be easily scaled up to an industrial application from a laboratory application (Borba et al., 2008).

For adsorption in continuous flow mode, fixed bed column are commonly utilized. The driving force of adsorption process is the existence of a gradient concentration which then favours mass transfer to occur. Breakthrough curves, which is a representation of pollutant effluent concentration against time profile in a fixed bed column, are usually used to characterize the continuous sorption process (de Franco et al., 2018). The design

and fixed bed column optimization commonly involve mathematical models. These mathematical models are then used for the description and prediction of the experimental data and is a beneficial tool which can be utilized for the scale up purposes (Borba et al., 2008).

1.2 Problem Statement

Activated carbon has been widely used as an adsorbent for the removal of pollutant by using adsorption process. With the ever increasing modernization, a lot of industries start blooming especially the manufacturing industry. Manufacturing industry in Malaysia includes production of chemicals and petroleum products, electric and electronic product, wood products, textiles, apparel and footwear, construction-related materials, transport equipment and food and beverages (Wan, 2016). The increasing activity of these industries lead to major damage and impact to the environment.

One of the increasingly threatening pollution is the heavy metal pollution (Nazli and Hashim, 2010). Thus, a lot of studies had been done in order to find the most efficient way to remove heavy metal from the wastewater. One of the few methods that are being studied is the usage of activated carbon to remove these heavy metals. Regeneration step is done after the adsorption process so that the heavy metal can be reuse or disposed of in a more efficient and environment friendly method. However, some heavy metal may still be left in the activated carbon.

In that case, to prevent from disposing the activated carbon and it being wasted, the activated carbon can be reuse for adsorption of other water pollutant. Since textile industry also seems to display an increase in the scheduled waste generated in Malaysia, so the adsorbate to be is dye (Yeoh et al., 1993).

Thus, the optimum impregnation ratio of heavy metal and activated carbon should be determined so that the maximum adsorption capacity can be achieved.

1.3 Research Objectives

The objective of this research are:

- i. To study the effect of modification of activated carbon on the performance of adsorption bed.
- ii. To determine the effects of different parameters on the adsorption capacity of the bed.
- iii. To determine the suitable kinetic models to be used to construct the dynamic adsorption curve.

1.4 Research Scope

In this study, coconut shell activated carbon was impregnated with chromium at specific impregnation ratio which has been predetermined. After impregnation, the adsorbent were characterized using Fourier Transform Infrared (FTIR) Spectroscopy analysis and Brunauer-Emmett-Teller (BET) analysis. The characterization was done in order to study the surface area and surface chemistry of the adsorbent. Then, the column adsorption study were performed using the adsorbent and Acid Yellow 17 as the adsorbate. Through this, adsorption capacity of the adsorbent bed can be obtained. The breakthrough curves for the adsorption of adsorbate was analysed using several kinetic models. By using error analysis method, the best dynamic model curve was determined.

1.5 Organization of Thesis

This thesis consist of five main chapters. The contents for each chapter is explained in the following:

Chapter 1 (Introduction) introduces the background and the significance of conducting the study of dynamic adsorption of dye on the adsorbate, the problem statement, research objectives and the research scope.

Chapter 2 (Literature Review) covers the literature review related to this study. This chapter introduces about the adsorbate which is dye and also the technologies available for dye removal. Next, more insights were given for the adsorption process as well as the adsorbent. Finally, the breakthrough curves for the adsorption of adsorbate was discussed and the kinetics model that is used was introduced.

Chapter 3 (Materials and Methods) presents the materials required and methodology steps done in this study. The materials and equipment used, adsorbent characteristics, characterization steps, experimental procedure and breakthrough study of adsorption in fixed bed column were also described in this chapter.

Chapter 4 (Results and Discussion) presents the experimental results and discussions for the experimental data obtained. The characterization of adsorbent results were interpreted and discussed. The effect of different parameters on the adsorption of dye on modified activated carbon and the breakthrough curve studies were discussed and provided in this chapter.

Chapter 5 (Conclusion and Recommendations) concludes the overall findings and discussions done in this research study. Related recommendations was also given in this chapter for further studies on this research topic.

CHAPTER 2

LITERATURE REVIEW

2.1 Dyes

Colour was the first contaminant to be identified in wastewater and it has to be removed before the wastewater can be discharged into open waterbodies or land. The presence of even a very small amount of dyes in water (less than 1 ppm for some dyes) can be extremely visible. These can affect the water transparency, aesthetic value and gas solubility in rivers, lakes and other waterbodies sources (Banat et al., 1996). Until the late nineteenth century, all colorants were obtained from nature especially from plants or insects and molluscs. In order to produce a small amount of impure dye, extensive amount of raw materials were required and the process was intensive. In 1856, when Perkin discovered the first synthetic dye, mauveine, dyes were manufactured in large scales using chemical intermediates (Christie, 2007). These days, many industries use dyes to colour their products. These industries include dyestuffs, textile, paper and plastic. They also consume a considerable volume of water. As a consequence, substantial amount of coloured wastewater was produced (Crini, 2006).

2.1.1 Classification Systems for Dyes

Dyes can be classified by their chemical structure and their usage or application method. Classification by chemical structure is usually applied by practicing dye chemists where they use terms such as azo dyes, phthalocyanine dyes and anthraquinone dye. Dye users and dye technologist usually used the dye classification by their usage and application method. For example, azo disperse dye for polyester and phthalocyanine reactive dye for cotton (Hunger, 2007). Dye can also be classified according to their

solubility. For instance, soluble dyes include direct, basic and reactive dyes while insoluble dyes include azoic, vat and disperse dyes (Gupta and Suhas, 2009).

It is, however, advantageous to classify dye based on the application before considering the chemical structure. This is because the dye nomenclature for classification by chemical structure is very complex. Besides, it is worth to point that the Colour Index (C.I) also adopt the system of classification based on the application of dyes (Gupta and Suhas, 2009). Table 2.1 shows the classification of dyes according to their usage and is arranged according to the C.I. classification.

Table 2.1: Usage classification of dyes (Hunger, 2007).

Class	Principal substrates	Method of application	Chemical types
Acid	nylon, wool, silk, paper, inks, and leather	usually from neutral to acidic dyebaths	azo (including premetallized), anthraquinone, triphenylmethane, azine, xanthene, nitro and nitroso
Basic	paper, polyacrylonitrile, modified nylon, polyester and inks	applied from acidic dyebaths	cyanine, hemicyanine, diazahemicyanine, diphenylmethane, triarylmethane, azo, azine, xanthene, acridine, oxazine, and anthraquinone
Direct	cotton, rayon, paper, leather and nylon	applied from neutral or slightly alkaline baths containing additional electrolyte	azo, phthalocyanine, stilbene and oxazine

Table 2.1: *continued*

Disperse	polyester, polyamide, acetate, acrylic and plastics	fine aqueous dispersions often applied by high temperature pressure or lower temperature carrier methods; dye may be padded on cloth and baked on or thermofixed	azo, anthraquinone, styryl, nitro and benzodifuranone
Reactive	cotton, wool, silk and nylon	reactive site on dye reacts with functional group on fiber to bind dye covalently under influence of heat and pH (alkaline)	azo, anthraquinone, phthalocyanine, formazan, oxazine and basic
Solvent	plastics, gasoline, varnishes, lacquers, stains, inks, fats, oils and waxes	dissolution in the substrate	azo, triphenylmethane, anthraquinone and phthalocyanine
Sulfur	cotton and rayon	aromatic substrate vatted with sodium sulphide and reoxidized to insoluble sulfur-containing products on fiber	interdeterminate structure
Vat	cotton, rayon and wool	water-insoluble dyes solubilized by reducing with sodium hydrogensulfite then exhausted on fiber and reoxidized	anthraquinone (including polycyclic quinone) and indigoids

2.1.2 Reactive Dyes

In 1954, it was reported that Rattee and Stephen had revealed that dyes which contain the 1,3,5-triazinyl group with chlorine substituents can react with cellulosic fibers under mild alkaline conditions without significant observable degradation of the fiber. The first series of water- soluble reactive dyes for cellulosic fibers named Procion MX were launched by ICI just two years after the discovery by Rattee and Stephen. According to Christie (2001), the development of reactive dyes can be perceived as the most important innovation in the technology of textile dyeing of the twentieth century.

Reactive dye are water soluble, anionic, coloured compound. After reactive dyes are applied on the textile fiber, they are invoke to react chemically with the fiber and covalent bond are formed between a carbon atom of the dye molecule and an oxygen, nitrogen or sulfur atom of a hydroxyl, amino or thiol group of the polymer that make up the fiber. Reactive dyes which are applied to the textile material can withstand from removal due to the strong covalent bond. This shows excellent washfastness properties.

Reactive dyes have develop steadily through the years to be the most important industrial class of dyes for the application to cellulosic fibers. Despite the decline of cotton derived textiles due to global production of synthetic fibers, production of reactive dyes for cellulose continues to increase (Christie, 2001).

2.1.3 Technologies for Dye Removal

The wastewater generated by textile wet-processing industries mainly originated from the washing and bleaching of natural fibers as well as the dyeing and finishing steps. There are a lot of variety of dyes, fibers and finishing products in use, which in turns generate wastewaters which have great chemical complexity (Vandevivere et al., 1998). Therefore, these wastewater need to be treated in order to remove the dyes.

Table 2.2 shows several methods and technologies for the removal of dyes from wastewaters. The technologies can be divided into three categories namely biological, chemical and physical (Robinson et al., 2001).

Table 2.2: Principal existing and emerging processes for dyes removal (Crini, 2006).

	Technology	Advantages	Disadvantages
Conventional treatment processes	Coagulation	Simple, economically feasible	High sludge production, handling and disposal problems
	Flocculation		
	Biodegradation	Economically attractive, publicly acceptable treatment	Slow process, necessary to create an optimal favourable environment, maintenance and nutrition requirements
	Adsorption on activated carbons	The most effective adsorbent, great capacity, produce a high-quality treated effluent	Ineffective against disperse and vat dyes, the regeneration is expensive and results in loss of the adsorbent, non-destructive process
Established recovery processes	Membrane separations	Removes all dye types, produce a high-quality treated effluent	High pressures, expensive, incapable of treating large volumes
	Ion-exchange	No loss of sorbent on regeneration, effective	Economic constraints, not effective for disperse dyes
	Oxidation	Rapid and efficient process	High energy cost, chemicals required

Table 2.2: *continued.*

Emerging removal processes	Advanced oxidation process	No sludge production, little or no consumption of chemicals, efficiency for recalcitrant dyes	Economically unfeasible, formation of by-products, technical constraints
	Selective bioadsorbents	Economically attractive, regeneration is not necessary, high selectivity	Requires chemical modifications, non-destructive process
	Biomass	Low operating cost, good efficiency and selectivity, no toxic effect on microorganisms	Slow process, performance depends on some external factors (pH, salts)

2.1.3 (a) Biological Treatments

Comparing to physical and chemical treatment, biological treatment is most often the economical choice. Biodegradation methods includes fungal decolourization, microbial degradation and bioremediation systems (Crini, 2006). However, the application is always limited due to technical restrictions. The limitations may include large land area requirement, less flexibility in design and sensitivity towards diurnal variation (Bhattacharyya and Sarma, 2003), as well as unsatisfactory colour elimination with current conventional biodegradation process (Robinson et al., 2001).

2.1.3 (b) Chemical Methods

Chemical methods are often expensive and include coagulation or flocculation combined with flotation and filtration, electroflotation, conventional oxidation methods by oxidizing agents (ozone) and irradiation. Accumulation of concentrated sludge may occur although dyes are removed and this leads to disposal problems. Secondary pollution

problem may also occur due to extreme chemical usage. New, rising techniques known as advanced oxidation processes are efficient for contaminated water treatment as powerful oxidizing agents is applied to degrade pollutant. However, this technique is very commercially unattractive and costly because of high electrical energy and chemical reagent consumptions (Crini, 2006).

2.1.3 (c) Physical Methods

Some of the very commonly used physical methods are membrane-filtration processes (nanofiltration and reverse osmosis) and adsorption techniques. Membrane processes has a huge setback having limited lifetime due to membrane fouling. So, the cost or periodic replacement must be included into the economic viability of this method. Adsorption, especially liquid phase adsorption can be considered the more popular and attractive alternative to treat contaminated water. This is because of the inexpensive sorbent and also no additional pre-treatment step is required before application. Thus, it can be concluded that adsorption is found to be the superior dye removal technique in terms of initial cost, simplicity in design, ease of operation, flexibility and insensitivity to toxic pollutants (Crini, 2006).

2.2 Adsorbents

Adsorbents are characterize by the amount of adsorbate that the adsorbent can accumulate. The amount of adsorbate can be calculated by using the adsorption isotherms (Gupta and Suhas, 2009). Adsorbents that are generally used for dye removal in wastewater treatment are activated alumina, silica gel, zeolites and activated carbon (Gupta and Suhas, 2009).

2.2.1 Activated Carbon

Activated carbon has been validated to be an effective absorbent which can be used to remove an assortment of organic and inorganic pollutants which are dissolved in aqueous media (Yin et al., 2007). Activated carbon can be produced from any type of carbonaceous materials. For activated carbon that is used commercially, the common raw materials are coal, lignite, coconut shell wood and agricultural by-products such as cattail leaves and cotton stalks (Anisuzzaman et al., 2015). Activated carbon is a carbon-based materials. It contains a well-developed internal micro pore structure and is a highly porous adsorbent with high surface area (Bhatnagar et al., 2013, Momčilović et al., 2011). As a consequence, activated carbon displays a high efficiency for adsorption of compounds that have low molecular weight and low efficiency for adsorption of compounds with large molecules. So for larger sized compounds, materials or activated carbon with high mesopore contribution to the total pore volume of adsorbent is much favoured (Lorenc-Grabowska and Gryglewicz, 2007). Activated carbon, which is hydrophobic, has always been used as a versatile adsorbent (Jaroniec, 2003). Activated carbon also encompasses a wide range of surface functional group such as carboxyls, phenols, lactones, aldehydes, ketones, quinones, hydroquinones and anhydrides (Yin et al., 2007, Aygün et al., 2003). However, activated carbon consists of these functional groups being bonded to ‘fused’ aromatic rings. So they are foreseen to have a chemical properties similar to that in aromatic hydrocarbons (Chingombe et al., 2005). Besides that, these functional groups contributes to the acid-base character of the activated carbon (Aygün et al., 2003). Even so, it is now well approved that acid or base character of a carbon is dependent on the history of how it is prepared and the treatment conditions in which it was oxidised (Nevskaia et al., 1999). The electrical change on the surface functional groups of the activated carbon may also improve or decrease the adsorption of targeted molecules.

Adsorption will increase if the adsorbate and the carbon surface carry opposite charges (Aygün et al., 2003).

2.3 Adsorption

Adsorption is the enrichment of one or more of the components in the region between two bulk phases (interfacial layer). One of the phases involved needs to be a solid and the other is a liquid (Rouquerol et al., 2013). In an adsorption process, molecules or atoms or ions which is in a gas or liquid will diffuse to the surface of a solid, where they will bond with the solid surface or are held there by weak intermolecular forces. The solid material is referred as the adsorbent while the adsorbed solutes are adsorbate (Seader and Henley, 1998). The description for the adsorption process is illustrated in the Figure 2.1.

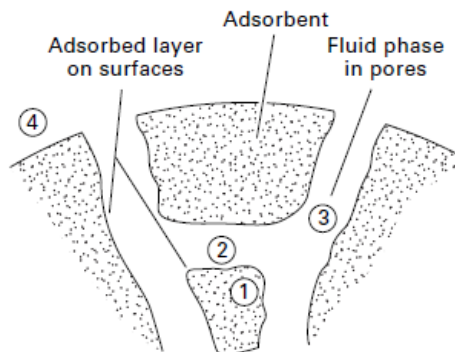


Figure 2.1: Adsorption operations with solid particle adsorbent (Seader and Henley, 1998).

Adsorption can be classified as physical adsorption or chemisorption depending on the type of forces between the fluid molecules and the solid molecules. Physical adsorption involves van der Waals forces or weak electrostatic forces formed between the adsorbent and adsorbate. Chemisorption involves the formation of chemical bonds between the adsorbent and adsorbate in monolayer (Seader and Henley, 1998). Desorption, on the other hand, denotes the converse process in which the molecules or

atoms or ions will transfer back from the solid surface to the gas or liquid phase (Dąbrowski, 2001). Molecules that are physisorbed can be desorbed and return to the fluid phase but chemisorbed molecules which undergoes reaction cannot be recovered by desorption (Rouquerol et al., 2013).

2.4 Adsorbents Used for Acid Dye Adsorption

2.4.1 Coconut Shell Activated Carbon

The activated carbon used is commercial activated carbon made from coconut shell. In Malaysia, coconut (*Cocos nucifera*) is one of the major crops with 142,000 ha of planted land. Hence, large amount of solid waste (mostly from the fiber and shell) are generated annually. Therefore, it is highly beneficial to convert coconut shell solid waste, which is composed mainly of lignin and cellulose with chemical composition similar to hard wood, into activated carbon considering that coconut shell is hard and is abrasion resistant (Mohd Din et al., 2009, Rodrigues and Pinto, 2007). Though not much studies were done on the removal of dye using coconut shell activated carbon, but there are studies conducted on the removal of heavy metals by coconut shell activated carbon such as lead (II) and Cr (VI) (Sekar et al., 2004, Babel and Kurniawan, 2004). Babel and Kurniawan (2004) concluded that coconut shell activated carbon is technically practical, economically viable and applicable for wastewater treatment especially in Cr (VI) removal. A study done by Kannan and Sundaram (2001) on the kinetics and mechanism of removal of methylene blue by adsorption shows that coconut shell activated carbon results in average relative adsorption capacity (Kannan and Sundaram, 2001).

2.4.2 Metal Impregnated Activated Carbon

There are several ways in which the AC can be modified in order to increase surface adsorption and removal capacity. Modifications include chemical treatment, physical treatment and also biological treatment. Acidic treatment, which is one of the chemical treatment for modification of AC, is one of the most studied technique (Yin et al., 2007). Acid treatment is generally applied to increase the acidic property (removing hydroxide groups on carbon surface) which will oxidize the porous carbon surface. Nitric acid and sulphuric acid are one of the few most widely researched acid for modifications of AC (Bhatnagar et al., 2013).

However, through the modernization of industry, chromium compounds is widely used. This results in chromium being discharged heavily to the environment. Chromium are used mainly in the electroplating of metal for corrosion resistance, production of steel, plastic coatings, leather tanning and finishing, photographic material, in pigments and for wood preservative (Mohan et al., 2006, Fahim et al., 2006). The discharge can cause serious damage and impact to the environment (Fahim et al., 2006). So there are a few studies done by other researchers in order to remove chromium from wastewater using the activated carbon (Fahim et al., 2006, Huang and Wu, 1977, Mohan et al., 2006, Monser and Adhoum, 2002, Mohanty et al., 2005).

The regeneration step needs to be able to separate the chromium and the activated carbon in order to be able to reuse the activated carbon. Some chromium may not be able to regenerate. So the chromium impregnated activated carbon can be used for dye removal.

2.5 Continuous Fixed Bed Adsorption Column

2.5.1 Adsorption capacity analysis

The breakthrough curve describes the performance of the fixed bed column. The breakthrough curve is expressed as the ratio of effluent dye concentration to the inlet dye concentration (C_t/C_0) as a function of time (Aksu and Gönen, 2004). The value of q_{total} for a given feed concentration and flow rate is equal to the area under the plot of the adsorbed AY 17 concentration C_{ad} ($C_{ad} = C_0 - C_t$) (mg/L) against t (min) and can be calculated from equation below:

$$q_{total} = \frac{Q}{1000} \int_{t=0}^{t=t_{total}} C_{ad} dt \quad (2.1)$$

The value of $q_{e(exp)}$ is calculated as the following:

$$q_{e,exp} = \frac{q_{total}}{x} \quad (2.2)$$

W_{total} is calculated from Eq.:

$$W_{total} = \frac{C_0 Q t_{total}}{1000} \quad (2.3)$$

Y is the ratio of the maximum capacity of the column (q_{total}) to the total amount of AY 17 sent to the column (W_{total}).

$$Y = \frac{q_{total}}{W_{total}} \times 100 \quad (2.4)$$

Prediction of the concentration-time profile or breakthrough curve for the effluent leads to a successful design of column adsorption process. Kinetics model are then used to express the dynamic process of the column mode (Han et al., 2009).

2.5.2 Column Dynamic Adsorption Model

The breakthrough curve and the adsorption capacity of the adsorbent for the selected adsorbate must be predicted when designing an adsorption column process. The

maximum sorption column capacity must also be determined (Chafi et al., 2016). Kinetic models are used to express mathematically the dynamic process of the column. The kinetic models are developed to evaluate the efficiency and the applicability of the column models for large scale operations. The Thomas, Yoon-Nelson, Adams-Bohart, and Bed Depth/ Service Time analysis Model was used in this study.

2.5.2 (a) Thomas Model

Thomas model assumes a plug flow behaviour in the bed and is one of the most widely used kinetic models for column adsorption studies (Chafi et al., 2016). It estimates the maximum phase concentration of adsorbate on adsorbent and the adsorption rate constant. The system is applicable where there are no external or internal diffusion resistance and axial dispersion (Jafari et al., 2017). The non-linear form of Thomas model is given as below:

$$\frac{C_t}{C_o} = \frac{1}{1 + \exp\left(\frac{K_{Th}q_0x}{Q} - K_{Th}C_0t\right)} \quad (2.5)$$

The linearized form of the model is given by:

$$\ln\left(\frac{C_o}{C_t} - 1\right) = \left(\frac{K_{Th}q_0x}{Q}\right) - (K_{Th}C_0t) \quad (2.6)$$

where K_{Th} is the Thomas rate constant ($\text{mL min}^{-1} \text{mg}^{-1}$), q_0 is the equilibrium adsorbate uptake (mg g^{-1}), x is the amount of adsorbent in the column (g), C_o is the inlet dye concentration (mg L^{-1}), C_t is the effluent dye concentration (mg L^{-1}), t is the contact time (min) and Q is the flow rate (mL min^{-1}). A non-linear plot of C_o/C_t against time (t) is to be employed to determine the values of K_{Th} and q_0 from the intercept and slope of the plot (Afroze et al., 2016).

2.5.2 (b) Yoon-Nelson Model

Yoon-Nelson model is a model that is developed based on the assumption that the rate of decrease in the probability of adsorption of adsorbate molecule is proportional to the probability of the adsorbate adsorption and the adsorbate breakthrough on the adsorbent (Chafi et al., 2016). The equation for the Yoon–Nelson model for a single component system is expressed as:

$$\frac{C_t}{C_0 - C_t} = \exp(K_{YN}t - K_{YN}\tau) \quad (2.7)$$

where K_{YN} is the Yoon and Nelson rate constant (min^{-1}), C_0 is the inlet or initial concentration (mg L^{-1}), C_t is the effluent dye concentration (mg L^{-1}), t is the breakthrough (sampling) time (min), and τ is the time required for 50 % adsorbate breakthrough (min). The plot of C_0/C_t versus sampling time (t) was constructed according to equation above and the value K_{YN} and τ can be determined (Jafari et al., 2017).

Based on the value of τ obtain, the adsorption capacity, q_{0YN} , was calculated using:

$$q_{0YN} = \frac{q(\text{total})}{x} = \frac{C_0 Q \tau}{1000x} \quad (2.8)$$

2.5.2 (c) Adams-Bohart Model

Adams-Bohart model is established by assuming that the adsorption rate is proportional to both the residual capacity of the adsorbent and the concentration of adsorbing species. This model is used to describe the initial part of the breakthrough curve (Han et al., 2009). The equation of Adams-Bohart Model used is as follows:

$$\frac{C_t}{C_0} = \exp\left(K_{AB}C_0t - K_{AB}N_0\frac{Z}{U}\right) \quad (2.9)$$

where, C_0 and C_t are concentrations initial and effluent dyes solution (mg L^{-1}), K_{AB} is the Adams-Bohart rate constant ($\text{L g}^{-1} \text{min}^{-1}$), N_0 is adsorption capacity of the adsorbent dependent on flow rate (mg L^{-1}), Z is the bed depth in the column (cm), t is time (min)

and U is the linear velocity (cm min^{-1}) calculated by dividing the flow rate by the column sectional area (Chafi et al., 2016).

2.5.2 (d) Bed Depth/ Service Time analysis (BDST) Model

BDST is a simple model predicting the relationship between bed depth, Z and the service time, t , in terms of process concentrations and adsorption parameters. The equation of BDST model can be expressed as follows (Han et al., 2009):

$$t = \left(\frac{N_0 Z}{C_0 U} \right) - \left(\frac{1}{K_a C_0} \right) \ln \left(\frac{C_0}{C_t} - 1 \right) \quad (2.10)$$

where C_t is the effluent concentration of solute in the liquid phase (mg L^{-1}), C_0 is the inlet solute concentration (mg L^{-1}), U is the influent linear velocity (cm min^{-1}), N_{BD} is the adsorption capacity (mg L^{-1}), K_{BD} is the rate constant in BDST model ($\text{L mg}^{-1} \text{min}^{-1}$), t is time (min), and Z is the bed height of column (cm).

A plot of t versus bed height Z should yield a straight line where N_{BD} and K_{BD} can be determined. A simplified form of the BDST model is as follows:

$$t = az + b \quad (2.11)$$

where slope, $a = N_{BD}/C_0 U$ and intercept, $b = -(1/K_{BD} C_0) \ln[(C_0/C_t) - 1]$.

2.5.3 Error Analysis

To determine the best fit of the kinetic expression or equilibrium of the experimental data, the value of coefficient of determination, r^2 was used (Kumar and Sivanesan, 2006). r^2 is defined as below:

$$r^2 = \frac{\sum (q_{e,cal} - q_{e,av})^2}{\sum (q_{e,cal} - q_{e,av})^2 + \sum (q_{e,cal} - q_{e,exp})^2} \quad (2.12)$$

Non-linear analysis optimization was applied to determine the isotherm parameter values in this study. In order to determine and evaluate the fit of the isotherm to the

experimental equilibrium data, the error function is needed for the optimization process (Gimbert et al., 2008). In this study, the error function that is applied is the sum of the squares of errors (SSE) and the equation is shown below:

$$SSE = \sum_{i=1}^n (q_{e,cal} - q_{e,av})_i^2 \quad (2.13)$$

2.6 Adsorbent reusability and regeneration

Granular activated carbon are usually used by packing into columns to adsorb pollutants from fluids passing through the columns. These packed columns can be used continuously until the AC is saturated with pollutants and the adsorption capacity will decrease drastically. When this happens, the packed AC must either be replaced or regenerated to restore the adsorptive capacity (Lu et al., 2011). Regeneration is normally cheaper than replacement according to Lu et al. (2011). Operational cost and product wastage can be minimize by regenerating the AC. Besides that, regeneration stabilize the saturated AC and this leads to the reduction in the amount solvents and adsorbents used in the process (El Gamal et al., 2018).

Numerous study done and methods has been developed previously to regenerate saturated AC. The summarized method for AC regeneration and reference for previous literature is shown in Table 2.3.

These methods are mostly efficient. However, these methods require the saturated AC to be removed from the packed column where regeneration is done off site and fresh or regenerated AC was replaced into the packed column. Thus, it can be said that chemical regeneration is an attractive method for regeneration of saturated AC (Lu et al., 2011). No carbon-attrition or pore structure degradation occurs when chemical regeneration was utilized.

Table 2.3: Summary of method for AC regeneration and reference for previous works.

Method	Reference
Steam regeneration	Urano et al., 1982
Thermal regeneration	San Miguel et al., 2001
Chemical regeneration	Lu et al., 2011
Microwave regeneration	Liu et al., 2004
Wet oxidation regeneration	Quesada-Peñate et al., 2012
Electrochemical regeneration	Brown and Roberts, 2007
Bio-regeneration	Aktaş and Çeçen, 2007

According to Lu et al. (2011), pure alcohol is more effective in desorbing yellow dyes at 25 °C compared to pure ketone with similar molecular weight. Dye is more soluble in alcohol because of the hydroxyl group in the alcohol so it is more easily desorbed by alcohol than ketone. The optimal concentration of ethanol for desorption of the AC saturated with yellow dye is 60 % v/v with optimal desorption efficiency (DE) of 84 %.

Table 2.4.

Table 2.4: Optimal concentration of various solvents to desorb the yellow dyes from the activated carbon (Lu et al., 2011).

Solvent	Yellow dye	
	Optimal concentration (%)	Optimal DE (%)
Methanol	100	85
Ethanol	60	84
Isopropanol	40	88
Ethylene glycol	80	67
Acetone	60	81
Formalin	100	74
Deionized water	-	18