

ONE-STEP SYNTHESIS AND ADSORPTIVE CHARACTERISTICS OF
COMPOSITE MAGNETIC ACTIVATED CARBON FOR RHODAMINE B REMOVAL

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of philosophy

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DECEMBER 2019

DEDICATION

This thesis is dedicated to my beloved parents and husband, for their love,
endless support and encouragement.

ACKNOWLEDGEMENT

First and foremost, I would like to express my very deep gratitude to Dr. Muhammad Abbas Ahmad Zaini, my supervisor. The supervision, advice and encourage that he gave truly help the progression and smoothness of this project to meet the objectives. His willingness to give his time so generously are much indeed appreciated. This thesis would not have been possible without his guidance and support.

In addition, my sincere thanks are extended to my parents and husband as well as all my family members. Their concerns and support had motivated me to complete this project within the time.

I wish to express my appreciation to colleagues and other relevant parties who have, directly and indirectly, contributed towards the completion of this project.

Last but not least, deepest thankful is expressed to Universiti Teknologi Malaysia for the support of UTM Zamalah scholarship.

ABSTRACT

Most of adsorption research has been carried out with dyes since their existence affects not only the quality of water but also changes the aquatic ecosystems as well as reduces the light penetration. Fine activated carbons are effective in dye adsorption, but they are extremely difficult to be separated from the solution when the carbons become exhausted. Magnetic activated carbon (MAC) has an advantage for the separation of spent activated carbon due to its excellent magnetic properties. However, complicated and multiple steps in the preparation process, and smaller adsorption capacity as compared to conventional activated carbon are the significant drawbacks of magnetic activated carbon. A simple synthesis method which simultaneously involves the activation and magnetization in a single step was introduced. This work was aimed at evaluating the adsorptive properties of composite magnetic activated carbons prepared from palm kernel shell. The activated carbons were prepared at various impregnation ratios of ZnCl_2 and FeCl_3 , activation temperatures ranging between 300 and 800°C and times of 1 to 3 h. ZMAC-2.5 prepared at 600°C and 2 h with impregnation ratios of $\text{ZnCl}_2:\text{FeCl}_3:\text{PKS} = 1.5:1.0:1.0$, which has BET surface area of 1775 m^2/g and mesoporosity of 93.8 % endowed a higher rhodamine B (RB) adsorption of 371 mg/g. For the purpose of comparison, MACs prepared by conventional magnetization methods and non-magnetic activated carbon (ZAC-1.5) were also employed in adsorption studies. MACs were characterized based on proximate analysis, elemental analysis, textural characteristics, chemical properties and magnetic properties. The batch adsorption was evaluated for equilibrium isotherm, kinetics and thermodynamics properties of RB by MACs. Langmuir isotherm model gave the best conformity of equilibrium data indicating a monolayer adsorption of RB onto activated carbons. The kinetics data were fitted better with pseudo-second-order model. The intraparticle diffusion and Boyd models revealed that both film and pore diffusion may be involved in the adsorption process, but none is the sole rate-limiting step. The positive values of enthalpy change and entropy change indicate that the adsorption process is endothermic and spontaneous at high temperature. The activation energy of 24.1 - 28.9 kJ/mol suggested that the adsorption of RB onto ZMAC-2.5 is a physisorption process. In fixed bed adsorption, the effect of initial RB concentration, flow rate and bed height were evaluated. The increase in initial concentration or flow rate, or the decrease in bed height could result in increasing adsorption capacity but decreasing breakthrough time. The column data fitted well with Thomas, Yoon-Nelson and Bohart-Adams models with high coefficient of determination (R^2) and low sum of squared errors (SSE). These models describe that the adsorption process is controlled by the interaction between RB molecules and ZMAC-2.5 surface. In hot water regeneration studies, ZMAC-2.5 showed a higher recovery than ZAC-1.5 for three consecutive regeneration cycles due to its magnetic properties. The optimum conditions were found to be 68.3 °C and 1 h to achieve the regeneration efficiency of 36.8 % and recovery of 93.7 %. Magnetic activated carbon showed a great potential to ease the separation of exhausted activated carbon using permanent magnet in wastewater treatment.

ABSTRAK

Kebanyakan kajian penjerapan telah dijalankan dengan pencilup kerana kewujudannya bukan hanya memberi kesan kepada kualiti air, ia juga mengubah ekosistem akuatik serta mengurangkan penembusan cahaya. Karbon teraktif halus berkesan dalam penjerapan pencilup, tetapi sukar dipisahkan daripada larutan apabila karbon menjadi tepu. Karbon teraktif magnet (MAC) mempunyai kelebihan untuk dipisahkan daripada air kerana sifat magnetnya yang sangat baik. Walau bagaimanapun, proses penyediaan yang rumit dan kapasiti penjerapannya yang lebih kecil berbanding karbon teraktif konvensional adalah kelemahan ketara bagi karbon teraktif magnet. Kaedah sintesis mudah yang melibatkan proses pengaktifan dan pemagnetan dalam satu langkah secara serentak telah diperkenalkan. Tujuan kajian ini adalah untuk menilai sifat-sifat penjerapan karbon teraktif magnet komposit yang disediakan daripada kulit isirung sawit. Penyediaan karbon teraktif dengan komposit $ZnCl_2$ dan $FeCl_3$ dilakukan pada pelbagai nisbah penepuan, suhu pengaktifan antara 300 hingga 800°C dan masa pengaktifan antara 1 hingga 3 jam. ZMAC-2.5 yang disediakan pada 600°C selama 2 jam dengan nisbah penepuan $ZnCl_2:FeCl_3:PKS=1.5:1.0:1.0$ mempunyai luas permukaan 1775 m²/g dan mesoporositi 93.8%, juga memberikan kapasiti penjerapan rhodamine B (RB) sebanyak 371 mg/g. Untuk tujuan perbandingan, MACs dengan kaedah pemagnetan lazim dan karbon teraktif bukan magnet (ZAC-1.5) juga disediakan dalam kajian penjerapan. MACs dicirikan berdasarkan analisis anggaran, analisis unsur, ciri-ciri tekstur, sifat-sifat kimia dan sifat-sifat magnet. Penjerapan berkelompok digunakan untuk mengkaji keseimbangan isoterma penjerapan, kinetik dan sifat termodinamik RB pada MACs. Model isoterma Langmuir memberikan pematuhan terbaik data keseimbangan, menunjukkan bahawa kehadiran penjerapan RB bersifat satu lapisan pada karbon teraktif. Data kinetik memberikan penyesuaian terbaik dengan model pseudo-tertib-kedua. Model resapan intrazarah dan model Boyd menunjukkan bahawa kedua-dua resapan filem dan liang mungkin berlaku dalam proses penjerapan, tetapi ia bukan langkah penghad kadar tunggal. Nilai-nilai positif perubahan entalpi dan perubahan entropi menunjukkan bahawa proses penjerapan adalah endotermik dan spontan pada suhu tinggi. Tenaga pengaktifan antara 24.1 hingga 28.9 kJ/mol mencadangkan bahawa penjerapan RB pada ZMAC-2.5 adalah proses penjerapan fizikal. Dalam penjerapan melalui turus lapisan tetap, kesan kepekatan RB awal, kadar alir dan ketinggian lapisan telah dikaji. Peningkatan kepekatan awal atau peningkatan kadar alir, atau penurunan ketinggian lapisan boleh mengakibatkan peningkatan kapasiti penjerapan tetapi menurunkan masa pintasan. Data turus proses penjerapan disesuaikan dengan model Thomas, Yoon-Nelson dan Bohart-Adam dengan pekali penentuan yang tinggi (R^2) dan jumlah ralat sisa kuadrat yang rendah (SSE). Kesesuaian model-model ini menerangkan bahawa proses penjerapan dikawal oleh tindak balas permukaan antara molekul RB dan permukaan ZMAC-2.5. Dalam penjana semula karbon teraktif dengan air panas, ZMAC-2.5 menunjukkan perolehan lebih tinggi berbanding dengan ZAC-1.5 bagi tiga kitaran penjana semula kerana sifat magnetnya. Keadaan operasi optimum adalah 68.3°C dan 1 jam untuk mencapai kecekapan penjana semula sebanyak 36.8% dan perolehan sebanyak 93.7%. Karbon teraktif magnet menunjukkan potensi besar bagi memudahkan pemisahan karbon teraktif tepu dalam rawatan air sisa.

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LIST OF ABBREVIATIONS

AA	-	Activating agent
AC	-	Activated carbon
ANOVA	-	Analysis of variance
BET	-	Brunauer–Emmett–Teller
BTC	-	Breakthrough curve
CAC	-	Commercial activated carbon
CHNOS	-	Carbon, hydrogen, nitrogen, oxygen and sulphur
C.I.	-	Colour Index
DBT	-	Dibenzothiophene
df	-	Degree of freedom
DMF	-	N,N-dimethylformamide
DR	-	Dubinín-Radushkevich
EDTA	-	Ethylenediaminetetraacetic acid
emu	-	Electromagnetic unit
FESEM	-	Field emission scanning electron microscope
FTIR	-	Fourier transform infrared spectroscopy
GAC	-	Granular activated carbon
IR	-	Impregnation ratio
IUPAC	-	International Union of Pure and Applied Chemistry
MAC	-	Magnetic activated carbon
MB	-	Methylene blue
MTZ	-	Mass transfer zone
MW	-	Molecular weight
PAC	-	Powdered activated carbon
pH _{PZC}	-	pH of the point of zero charge
PKS	-	Palm kernel shell

PKS-AC	-	Palm kernel shell based activated carbon
RB	-	Rhodamine B
RE	-	Regeneration efficiency
RP	-	Redlich-Peterson
RSM	-	Response surface methodology
SEM	-	Scanning electron microscope
SSE	-	Sum of squares of the residuals / sum of squared errors
SSR	-	Sum of squares due to regression
SST	-	Total sum of squares
TEM	-	Transmission electron microscope
TGA	-	Thermogravimetric analysis
VSM	-	Vibrating sample magnetometer
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

A	-	Frequency factor in Arrgenius equation
B	-	Magnetic flux density
b	-	Langmuir adsorption constant
c	-	Intercept to the y-axis
CO	-	Carbon monoxide
CO ₂	-	Carbon dioxide
C_{Ae}	-	Amount adsorbed on solid at equilibrium
C_d	-	Desorption concentration in solution
C_e	-	Equilibrium concentration of adsorbate solution
C_o	-	Initial concentration of adsorbate solution
C_r	-	Re-adsorption equilibrium concentration of adsorbate solution
C_t	-	Effluent concentration at time t
D_i	-	Effective diffusion coefficient
d_i	-	Internal diameter of column
D_p	-	Average pore diameter
d_p	-	Particle diameter
E	-	Mean free energy
E_a	-	Activation energy
FeCl ₃	-	Ferric chloride
Fe ²⁺	-	Ferrous ion
Fe ³⁺	-	Ferric ion
Fe ₃ O ₄	-	Magnetite
H	-	magnetizing force
h	-	Initial adsorption rate
HCl	-	Hydrochloride acid
H_C	-	Coercivity

H_2SO_4	-	Sulfuric acid
K, n	-	Freundlich constants
KOH	-	Potassium hydroxide
K_{ad}	-	Dubinin-Radushkevich isotherm constant
K_{BA}	-	Bohard-Adams rate constant
K_d	-	Distribution coefficient
k_d	-	Rate constant for intraparticle diffusion
K_{NY}	-	Yoon and Nelson rate constant
K_R, α_R	-	Redlich-Peterson isotherm constants
K_{TH}	-	Thomas rate constant
k_1	-	Rate constant of pseudo-first-order
k_2	-	Rate constant of pseudo-second-order
K_2CO_3	-	Potassium carbonate
L	-	Column length
m	-	mass
m_T	-	Total mass
M_R	-	Remanence magnetization
M_S	-	Saturation magnetization
$NaHCO_3$	-	Sodium bicarbonate
NaOH	-	Sodium hydroxide
Na_2CO_3	-	Sodium carbonate
N_o	-	Saturation concentration in bed
N_2	-	Nitrogen
Oe	-	Oersted
pH	-	Potential hydrogen
Q	-	Volumetric flow rate
q_d	-	Desorption amount
q_e	-	Equilibrium adsorption capacity
q_m	-	Maximum adsorption capacity

q_o	-	Maximum adsorption capacity in column adsorption
q_r	-	Equilibrium re-adsorption capacity
q_s	-	Dubinin-Radushkevich isotherm saturation capacity
q_T	-	Total bed capacity
q_t	-	Adsorption amount at time t
R	-	Gas constant (8.314 J/mol.K)
r	-	Radius of particle
R^2	-	Correlation coefficient
R_A^2	-	Adjusted correlation coefficient
R_A	-	Adsorbate removal percentage in column adsorption
R_L	-	Separation factor
S	-	Bed cross section area
S_{BET}	-	BET surface area
T	-	Temperature
t	-	time
t_b	-	Breakthrough time
t_E	-	Exhausted time
t_T	-	Total flow time
U_o	-	Superficial velocity
V	-	Volume
V_T	-	Total effluent volume
Z	-	Bed height
$ZnCl_2$	-	Zinc chloride
$\gamma\text{-Fe}_2\text{O}_3$	-	Maghemite
ΔG°	-	Gibbs energy
ΔH°	-	Enthalpy
ΔS°	-	Entropy
ε	-	Polanyi potential
λ_{max}	-	Maximum wavelength

- τ - Time required for 50% adsorbate breakthrough
- $\%_{micro}$ - Microporosity

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Activated carbon is widely used as an adsorbent for adsorption due to its large porous surface area, controlled pore structure and inert properties (See *et al.*, 2015; Lee and Zaini, 2015). Activated carbon can be prepared from a variety of raw materials, especially agricultural by-products such as coconut shells, corn cobs, apricot stone, and so on (Wan *et al.*, 2014; Rocha *et al.*, 2015; Albroomi *et al.*, 2017). These products are regarded as waste and can cause a serious disposal problem in some countries. Therefore, converting them into activated carbon is a feasible solution to the environmental problem.

Malaysia is one of the largest exporters of palm oil in the international market, which is expected about 20 million tonnes production of crude palm oil in the year 2017 (Intan, 2017). Besides the production of crude palm oil, a large amount of solid waste such as palm kernel shell is also an output from the palm oil industry. The amount of palm kernel shell generated is approximately 5.61 million annually (Rugayah *et al.*, 2014). The disposal of palm kernel shell contributes to a major economic and environmental issue. So, many research works have been carried out to study the conversion of this waste to activated carbon (Yacob *et al.*, 2009; Abechi *et al.*, 2013b; Ogunsile *et al.*, 2014; Lee and Zaini, 2015; Vincent *et al.*, 2016; Hidayu and Muda, 2016). Palm kernel shell is a promising candidate as the precursor of palm kernel shell because it can be activated into high porosity carbon in short time due to its high lignin content, low cellulose content and less fibrous structure (Lee and Zaini, 2015). In this study, palm kernel shell was used as the precursor of activated carbon.

Fine activated carbons are effective in adsorption of pollutants from water, but they are extremely difficult to be separated from the solution when the carbons become

exhausted. The traditional method for separating activated carbon is by filtration, so the filter blockage and the loss of carbon could be the significant regeneration issue, consequently, limiting its application in many fields (Jia *et al.*, 2011). Magnetic separation is considered as a rapid and effective technique for the removal of spent adsorbents from solution. Recently, great attention has been paid on the application of magnetic activated carbon in adsorption of organic pollutants, dyes and heavy metals such as trinitrophenol, methylene blue, arsenic(V) (Mohan *et al.*, 2011; Zhang *et al.*, 2015; Chomehoey *et al.*, 2013). However, the complication of multiple steps process for the preparations of magnetic activated carbon and deterioration in its specific surface area due to the pores blockage by the attachment of magnetic particles are the significant drawbacks that have to be resolved in order to apply magnetic activated carbon in wastewater treatment (Zaini *et al.*, 2017).

Most of the adsorption research has been carried out with organic compounds, including phenolic compounds and dyes, as well as heavy metals. Biodegradation potential of organic compounds is emerging treatment process. However, products of biodegradation are hazardous in some cases, while some organic materials are extremely recalcitrant to biodegradation. Adsorption is a potential technique for the removal of these organic substances from wastewater. Among all organic substances, dye, phenolic compounds and pesticides are highly received attention. Dyes wastewater is very difficult to approach with common methods due to unfavorable in terms of economy and technical since the dyes are recalcitrant molecules, resistant to aerobic digestion and stable to oxidizing agents (Gadd, 2009). Dye is a visible pollutant. Its existence affects not only the quality of surface water but also changes the aquatic ecosystems as well as reduces light penetration. Rhodamine B (RB) is a water-soluble cationic dye which is a common water tracer fluorescent to determine the rate and direction of flow and transport. RB is widely used in research because it has a strong absorption band in the visible region of 554 nm and high stability against pH change (Muhammad, 2014). RB irritates eyes and skin, respiratory as well as gastrointestinal tract (Rochart *et al.*, 1978). In this study, RB used as the specific adsorbate.

Batch adsorption study is usually applied to determine the effectiveness of the activated carbon and its adsorption mechanism by providing the maximum adsorption capacity, isotherm constants as well as kinetic properties. Although batch adsorption study provides useful data and parameters on the application of specific adsorbents for the removal of a specific pollutant, it is not sufficient while designing a treatment system for continuous operation. Therefore, a continuous adsorption study is necessary to be performed using columns and provide information for the industrial-scale (Afroze *et al.*, 2016; Albroomi *et al.*, 2017).

1.2 Problem Statement

Magnetic activated carbons have a great potential to be used in wastewater treatment due to their magnetic properties, which make more effective for the recovery of spent activated carbon from treated water. In recent years, magnetic activated carbons have been widely studied to remove organic and inorganic pollutants from wastewater. However, the preparations of magnetic activated carbon were often complicated which involved multiple steps process. Usually raw materials have to be converted into activated carbon by activation process, then followed by magnetization process including chemical coprecipitation and high-temperature impregnation. The attachment of magnetic particles could lead the blockage of the pores of activated carbon, thereby magnetic activated carbons have the drawbacks of smaller adsorption capacity compared to activated carbons as reported in previous studies.

A simple synthesis method was suggested which involves the activation and the magnetization process in a single step simultaneously opens up the possibility where the iron serves both as an activating agent and as a magnetic element in the preparation of magnetic activated carbon. However, low dye adsorption performance (22.31 mg/g) was reported since ferric chloride (FeCl_3) is not a strong activating agent (Cazetta *et al.*, 2016). According to Zhang *et al.* (2015), composite of magnetite (Fe_3O_4) and potassium carbonate (K_2CO_3) was used as activating agent in preparation of magnetic activated carbon, but lack of information was reported to show the improvement in adsorption performance of magnetic activated carbon. Zinc chloride

(ZnCl₂) is a strong activating agent and widely used in the preparation of activated carbons for research, while FeCl₃ have similar characteristics to ZnCl₂ in aqueous solution, which opens up the possibility of using composite of ZnCl₂ and FeCl₃ as activating agent in the preparation of activated carbon (Rufford *et al.*, 2010). Lee and Zaini (2017) reported that the specific surface area of composite ZnCl₂-FeCl₃ activated carbon (411 m²/g) is higher than that of ZnCl₂ activated carbon (395 m²/g) because FeCl₃ is also acted as mild activator in preparation of activated carbon.

In this study, composite magnetic activated carbon was prepared from palm kernel shell by one-step synthesis, making the procedure for the preparation of magnetic activated carbon simpler compared to the conventional multiple-step process. Composite of ZnCl₂ and FeCl₃ was used as the activating agent to minimize the drawbacks of poorer adsorption performance since ZnCl₂ can be served as strong activator whereas FeCl₃ can be served as magnetic element as well as mild activator. The magnetic activated carbons prepared by conventional magnetization methods and non-magnetic activated carbon (origin of magnetic activated carbon) were also employed for the comparison purpose. The adsorptive characteristics of composite magnetic activated carbon could show the improvement in pore development and adsorption performance without deterioration of magnetic properties in order to evaluate the potential of magnetic activated carbon in dye wastewater treatment.

1.3 Objectives

Four objectives of this research are stated below:

- i. To synthesize, optimize and characterize magnetic activated carbons from palm kernel shell by the composite of zinc and ferric chloride salts activation.
- ii. To establish the adsorptive properties of rhodamine B removal by activated carbons at different initial concentrations, pH, time intervals and temperatures.
- iii. To investigate the effect of bed height, flow rate and initial concentration on column adsorption studies.

- iv. To evaluate the regeneration of spent activated carbon using hot water.

1.4 Scope of Study

- i. To synthesize, optimize and characterize magnetic activated carbons from palm kernel shell by the composite of zinc and ferric chloride salts activation.

Palm kernel shell was used as the precursor in the synthesis of magnetic activated carbon. Impregnation was carried out using composites of metals chloride salts as activating agents, which were $ZnCl_2$ and $FeCl_3$ at various impregnation ratios from 1.0 to 2.5. The heating temperature and heating period were varied from 300 to 800 °C and 1 to 3 h, respectively. Response surface methodology (RSM) was used to optimize the operating condition for the preparation of activated carbons. Conventional methods for preparation of magnetic activated carbon including chemical coprecipitation and high-temperature impregnation were carried out with optimum impregnation ratio of activating agent.

Activated carbons were characterized based on proximate analysis, elemental analysis, specific surface area, morphology, surface functional group, Boehm titration, magnetic properties and X-Ray diffraction analysis.

- ii. To establish the adsorptive properties of pollutant removal by activated carbons at different initial concentrations, pH, time intervals and temperatures.

Rhodamine B was used as the adsorbate model. Four isotherm models which are Langmuir, Freundlich, Redlich-Peterson and Dubinin-Radushkevich were used to fit the adsorption data at initial concentrations from 5 to 800 mg/L. The rate of adsorption for three initial concentrations at time intervals from 0.17 to 72 h was evaluated using the pseudo-first-order equation, pseudo-second order equation, intraparticle diffusion model and Boyd model. The thermodynamics properties, name by Gibbs energy, ΔG° , enthalpy, ΔH° and entropy, ΔS° were investigated through

the effect of temperature on dye adsorption from 30 to 60 °C for the best-performed activated carbon.

- iii. To investigate the effect of bed height, flow rate and initial concentration on column adsorption studies.

The effect of initial concentration of rhodamine B from 5 to 15 mg/L, flow rate from 16 to 28 mL/min and bed height from 2 to 6 cm were investigated on column adsorption of rhodamine B by the best-performed activated carbon and analysed based on three models, which are Thomas, Yoon-Nelson and Bohart-Adams models.

- iv. To evaluate the regeneration of spent activated carbon using hot water.

The regeneration study was performed using hot water at various temperatures and time for three consecutive adsorption-desorption cycles to determine the regeneration efficiency and recovery of the best-performed activated carbon. Response surface methodology (RSM) was used to optimize the operating condition for regeneration.

1.5 Significance of Study

This study is carried out to give a further understanding of the contribution of agricultural waste activated carbon to wastewater treatment. The issues related to palm kernel shell management can be reduced if it is converted into activated carbon. The simplicity of magnetic separation technique makes the suitability of activated carbon for wider applications. This study also proposes an environmentally friendly approach for regeneration method of spent activated carbon.

1.6 Thesis Outline

This thesis composed of five chapters that embody the research works in sequential order. Firstly, Chapter 1 introduces the research background, problem statement, objectives, scopes of study, significance of study as well as thesis outline. Then, Chapter 2 consists of an overview of previous research on activated carbon, magnetic activated carbon, dyes and environment, adsorption, regeneration of activated carbon as well as response surface methodology. Next, Chapter 3 includes all the materials used and covers the procedures and methods involves in this study, which are synthesis, optimization and characterizations of magnetic activated carbons, adsorption studies in batch and column mode as well as approach of regeneration studies. After that, Chapter 4 presents the results and the analyzed experimental data with discussion. Finally, Chapter 5 concludes the findings according to the objectives in this study and provides recommendations for future work.

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