ADSORPTION OF RHODAMINE B BY METALS CHLORIDE-ACTIVATED CASTOR BEAN RESIDUE CARBON

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ADSORPTION OF RHODAMINE B BY METALS CHLORIDE-ACTIVATED CASTOR BEAN RESIDUE CARBON

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Special dedicated to my beloved father and mother.

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ABSTRACT

Zinc chloride ($ZnCl_2$) is a well-known pollutant which is toxic to the aquatic organisms. A study of adsorption of rhodamine B was conducted to investigate the performance of metals chloride activated carbon prepared from castor bean residue. Rhodamine B was selected as the model dye due to its high stability with change in pH and hazardous properties. Castor bean residue is suitable to be used as precursor to replace conventional activated carbon due to its low cost and high carbon content. The preparation of activated carbons was conducted through impregnation with $ZnCl_2$, potassium chloride, magnesium chloride, ferric chloride and metals chloride composite at various impregnation ratios from 0.5 to 2.5. Activated carbons were characterized based on proximate analysis, elemental analysis, textural characteristics and chemical properties. The adsorption data were analysed using isotherm models, kinetics models and thermodynamics properties. The regeneration of activated carbon was carried out by hot water and irradiated water at three regeneration cycles. The specific surface area of activated carbons of ratio 1.0 are in descending order of potassium chloride (KCBR-1.0), ferric-zinc chloride (FZCBR), magnesium-zinc chloride (MZCBR), zinc chloride (ZCBR-1.0), ferric chloride (FCBR-1.0), potassium-zinc chloride (KZCBR), magnesium chloride (MCBR-1.0). ZCBR-1.0 demonstrated a greater rhodamine B adsorption of 175 mg/g compared to the other activated carbons counterparts. Nevertheless, the composite activated carbons, MZCBR and FZCBR displayed adsorptive capacity of 114 and 115 mg/g, respectively, which indicates the mixtures of less hazardous metal chloride salts as the promising activating agents. The adsorption capacity of rhodamine B by activated carbons of ratio 1.0 are in descending order of ZCBR-1.0, FZCBR, MZCBR, FCBR-1.0, MCBR-1.0, KCBR-1.0, KZCBR. Adsorption mechanism of ZCBR-1.0 obeyed Langmuir isotherm and pseudo-second-order kinetics model. The rate-limiting step in the adsorption of rhodamine B is film diffusion. The positive values of enthalpy change and entropy change indicate of that the adsorption process is endothermic and spontaneous at high temperature. Hot water regeneration onto rhodamine B loaded activated carbon showed a better performance with 37.7 % regeneration efficiency and 34.4% recovery.

ABSTRAK

Zink klorida ($ZnCl_2$) adalah pencemar toksik kepada organisma akuatik. Satu kajian penjerapan *rhodamine B* telah dijalankan untuk mengkaji prestasi karbon teraktif daripada sisa kacang kastor dengan pengaktifan logam klorida. *Rhodamine B* dipilih sebagai model pencelup kerana kestabilannya yang tinggi terhadap perubahan pH dan sifat-sifat berbahaya. Sisa kacang kastor sesuai digunakan sebagai prapenanda untuk menggantikan karbon teraktif lazim kerana ia murah dan mempunyai kandungan karbon yang tinggi. Penyediaan karbon teraktif dikendalikan melalui impregnasi dengan $ZnCl_2$, kalium klorida, magnesium klorida, ferik klorida dan komposit logam klorida pada pelbagai nisbah impregnasi dari 0.5-2.5. Karbon teraktif dicirikan berdasarkan analisis hampiran, analisis unsur, ciri-ciri tekstur dan sifat-sifat kimia. Data penjerapan dianalisis dengan model isoterma, model kinetik dan sifat termodinamik. Penjanaan semula karbon teraktif dengan air panas dan air teriradiasi dijalankan pada tiga kitaran penjanaan semula. Luas permukaan tentu karbon teraktif dengan nisbah 1.0 adalah mengikut tertib menurun kalium klorida (KCBR-1.0), ferik-zink klorida (FZCBR), magnesium-zink klorida (MZCBR), zink klorida (ZCBR-1.0), ferik klorida (FCBR-1.0), kalium-zink klorida (KZCBR), magnesium klorida (MCBR-1.0). ZCBR-1.0 memberikan prestasi penjerapan *rhodamine B* lebih tinggi dengan 175 mg/g berbanding dengan karbon teraktif yang lain. Walaubagaimanapun, karbon teraktif komposit, MZCBR dan FZCBR masing-masing memberikan kapasiti jerapan 114 and 115 mg/g yang menunjukkan pengaktifan campuran garam logam klorida kurang berbahaya sebagai agen pengaktifan yang berpotensi. Kapasiti penjerapan *rhodamine B* oleh karbon teraktif dengan nisbah 1.0 adalah mengikut tertib menurun ZCBR-1.0, FZCBR, MZCBR, FCBR-1.0, MCBR-1.0, KCBR-1.0, KZCBR. Mekanisma penjerapan ZCBR-1.0 mematuhi model isoterma *Langmuir* dan model kinetik pseudo-tertib kedua. Langkah kadar-penghad dalam penjerapan *rhodamine B* ialah resapan filem. Nilai-nilai positif perubahan entalpi dan perubahan entropi menunjukkan bahawa proses penjerapan adalah endotermik dan spontan pada suhu tinggi. Penjanaan semula karbon teraktif terjerap *rhodamine B* menggunakan air panas menunjukkan prestasi lebih baik dengan kecekapan penjanaan semula 37.7% dan perolehan 34.4%.

TABLE OF CONTENT

CHAPTER	TITLE	PAGE
	DECLARATION	ii
	DEDICATION	iii
	ACKNOWLEDGEMENT	iv
	ABSTRACT	v
	ABSTRAK	vi
	TABLE OF CONTENT	vii
	LIST OF TABLES	xi
	LIST OF FIGURES	xiii
	LIST OF ABBREVIATIONS	xv
	LIST OF APPENDICES	xvii
1	**INTRODUCTION**	1
	1.1 Research Background	1
	1.2 Problem Statement	3
	1.3 Objective	4
	1.4 Scope of Study	4
	1.5 Significant of Study	5

2	**LITERATURE REVIEW**		**6**
	2.0	Introduction	6
	2.1	Dyes	7
		2.1.1 Applications and Implications of Dyes	7
		2.1.2 Rhodamine B	11
		2.1.3 Treatment Methods	13
	2.2	Adsorption	16
		2.2.1 Types of Adsorption	17
		2.2.2 Adsorbents for Rhodamine B Removal	18
		2.2.3 Factors Affecting Adsorption of Rhodamine B	19
		2.2.4 Adsorption Modelling	22
		2.2.4.1 Equilibrium Isotherm	22
		2.2.4.2 Kinetic Models	25
		2.2.4.3 Thermodynamic Properties	27
	2.3	Activated Carbon	29
		2.3.1 Characterization of Activated Carbon	30
		2.3.2 Precursors of Activated Carbon	32
		2.3.3 Activation Methods	35
		2.3.4 Metals Chloride Salts as Activating Agents	36
		2.3.4.1 Metals Chloride Activation	36
		2.3.4.2 Hazardous Properties of Metals Chloride Salts	43
		2.3.5 Adsorption of Rhodamine B onto Activated Carbon	50
		2.3.6 Regeneration of Activated Carbon	51
	2.4	Summary	55

3	**METHODOLOGY**	56
	3.0 Introduction	56
	3.1 Materials	56
	3.2 Preparation of Castor bean Residue Activated Carbon (CBR-AC)	57
	3.3 Characterization of CBR-ACs	60
	3.3.1 Proximate Analysis of CBR	60
	3.3.2 Elemental Analysis (CHNOS)	61
	3.3.3 Textural Characterizations	62
	3.3.3.1 Specific Surface Area	62
	3.3.3.2 Morphology	64
	3.3.4 Chemical Properties	64
	3.3.4.1 pH of the Point of Zero Charge	65
	3.3.4.2 Surface Functional Groups	66
	3.4 Adsorptive Analysis	67
	3.4.1 Equilibrium Isotherm	68
	3.4.2 Kinetics Study	69
	3.4.3 Thermodynamics Properties	70
	3.5 Regeneration Procedures	70
4	**RESULTS AND DISCUSSIONS**	73
	4.0 Introduction	73
	4.1 Characterization of Castor Bean Residue Activated Carbons	73
	4.1.1 Proximate Analysis	74
	4.1.2 Elemental Analysis	77
	4.1.3 Textural Characteristics	78
	4.1.3.1 Morphology	78

4.1.3.2 Specific Surface Area and Porosity. 81

4.1.4 Chemical Properties 85

4.2 Adsorptive Analysis 92

4.2.1 Equilibrium Adsorption 92

4.2.1.1 Effect of Initial Dye Concentration 92

4.2.1.2 Isotherm Models 95

4.2.1.3 Effect of Solution pH 100

4.2.2 Adsorption Kinetics 102

4.2.2.1 Effect of Contact Time 102

4.2.2.2 Kinetics Models 104

4.2.3 Adsorption Thermodynamics 109

4.2.3.1 Effect of Temperature 109

4.2.3.2 Thermodynamics Properties 110

4.3 Regeneration Analysis 112

4.3.1 Recovery 112

4.3.2 Regeneration Efficiency 113

5 CONCLUSION AND RECOMMENDATIONS 117

5.1 Conclusion 117

5.2 Recommendations 118

REFERENCES 120

LIST OF PUBLICATIONS 143

Appendices A & B 144-147

LIST OF TABLES

TABLE NO.	TITLE	PAGE
2.1	Classification of dyes by application	8
2.2	Chemical properties of rhodamine B	12
2.3	Advantages and limitations of some treatment processes for dyes removal	14
2.4	Differences between physisorption and chemisorption	18
2.5	Some adsorbents for the removal of rhodamine B	19
2.6	Effect of increasing parameters on the adsorption of rhodamine B	22
2.7	Some well-known characterization method for AC	31
2.8	Composition of waste cakes	34
2.9	Chemical activation for waste cakes	35
2.10	Summary of metals chloride activation in recent literature	39
2.11	R-phrases based hazard rating	44
2.12	Effects of metals chloride salts to human and the environment	46
2.13	Maximum monolayer adsorption capacity of rhodamine B by various ACs	50
2.14	Regeneration of methylene blue-loaded ACs	54
3.1	Designation of activated carbons	59
4.1	Proximate analysis of CBR in this study and previous studies	75
4.2	Elemental analysis	77
4.3	EDX analysis of CBR char	81
4.4	Characteristics and possible surface functional groups of raw CBR	88
4.5	Functional groups of CBR char and ACs	90

4.6	pH_{PZC} and concentration of surface functional groups of some ACs	91
4.7	Langmuir and Freundlich isotherm constants	96
4.8	RP constants for RB adsorption by ACs	98
4.9	DR constants for RB adsorption by ACs	99
4.10	Kinetics constants of Pseudo-first-order and Pseudo-second-order models	105
4.11	Kinetics constants of intraparticle diffusion and Boyd's models	107
4.12	Thermodynamics parameters of ZCBR-1.0	111
4.13	Equilibrium adsorption of RB by ZCBR-1.0 at desorption-readsorption cycles with C_0 of 400 ppm	114

LIST OF FIGURES

FIGURE NO.	TITLE	PAGE
2.1	Structure of rhodamine B	12
2.2	Equilibrium adsorption curve	20
2.3	Rate of adsorption curve	21
2.4	Effect of impregnation ratio on production of AC	37
3.1	Procedures of activated carbons preparation	58
3.2	Characterization of activated carbons	60
3.3	Curve of final solution pH versus initial solution pH	65
3.4	Procedures of adsorptive analysis	67
3.5	Regeneration procedures of AC	71
4.1	TGA curve of CBR	74
4.2	Yield of ACs according to impregnation ratio of activating agents	76
4.3	FESEM image of CBR char at 2000 times magnification	78
4.4	FESEM image of ZCBR-1.0 at 2000 times magnification	79
4.5	FESEM image of FZCBR at 2000 times magnification	80
4.6	FESEM image of MZCBR at 2000 times magnification	80
4.7(a)	BET surface area and microporosity of ZCBR series	82
4.7(b)	BET surface area and microporosity of KCBR series	82
4.8	BET surface area of char and metals chloride-ACs at impregnation ratio of 1.0	83
4.9	Average pore diameter of ACs against ion radius of metals chloride	84
4.10	FTIR spectrum of raw CBR	87
4.11	FTIR spectra of CBR char and some ACs	89
4.12	Effect of initial concentration on the RB adsorption by ACs with impregnation ratio of 1.0	93

4.13	Effect of initial concentration on the RB adsorption by ZCBR series with different impregnation ratios	93
4.14	Effect of initial concentration on the RB adsorption by KCBR series with different impregnation ratios	94
4.15	Adsorption of RB by ZCBR-1.0 at various initial pH of RB solution	100
4.16	RB in cationic (a), lactonic (b) and zwitter-ionic (c) conformations	101
4.17	Effect of contact time on the RB adsorption by ACs with impregnation ratio of 1.0 at maximum initial concentrations	102
4.18	Effect of contact time on the RB adsorption by ZCBR series with different impregnation ratios	103
4.19	Effect of contact time on the RB adsorption by KCBR series with different impregnation ratios	103
4.20	Intraparticle diffusion model for ZCBR series at various initial concentrations	108
4.21	Adsorption capacity of ZCBR-1.0 at various temperature and initial concentration	109
4.22	Recovery of regenerated ZCBR-1.0	113
4.23	N_2 adsorption-desorption isotherm of ZCBR-1.0	114
4.24	Regeneration efficiency of dye-loaded ZCBR-1.0	115

LIST OF ABBREVIATIONS

AC	–	Activated carbon
BET	–	Brunauer, Emmett and Teller
C	–	Carbon
$CaCl_2$	–	Calcium chloride
CBR	–	Castor bean residue
CBR-AC	–	Castor bean residue activated carbon
CO	–	Carbon monoxide
CO_2	–	Carbon dioxide
$CuCl_2$	–	Cupper chloride
C_e	–	Equilibrium concentration
C_o	–	Initial concentration
EDX	–	Energy-Dispersive x-ray
$FeCl_2$	–	Ferrous chloride
$FeCl_3$	–	Ferric chloride
Fe^{3+}	–	Ferric ion
Fe_2O_3	–	Ferric oxide
FESEM	–	Field Emission Scanning Electron Microscope
FTIR	–	Fourier Transform Infrared Spectroscopy
HCl	–	Hydrochloride acid
HR	–	Hazard rating
H^+	–	Hydrogen ion
H_2O_2	–	Hydrogen peroxide
IUPAC	–	International Union of Pure and Applied Chemistry
KCl	–	Potassium chloride
KOH	–	Potassium hydroxide
K^+	–	Potassium ion

K_2CO_3	–	Potassium carbonate
LC	–	Lethal concentration
$MgCl_2$	–	Magnesium chloride
MgO	–	Magnesium oxide
Mg^{2+}	–	Magnesium ion
NaCl	–	Sodium chloride
O	–	Oxygen
OH^-	–	Hydroxide ion
$PbCl_2$	–	Lead chloride
pH	–	Potential hydrogen
pH_{PZC}	–	Point of zero charge
Q_e	–	Equilibrium dye concentration on the adsorbent
Q_{max}	–	Maximum adsorption capacity
R	–	Gas constant
RB	–	Rhodamine B
RE	–	Regeneration efficiency
R^2	–	Correlation coefficient
R_L	–	Equilibrium parameter
t	–	Time
T	–	Absolute temperature
TGA	–	Thermogravimetric analysis
UTM	–	Universiti Teknologi Malaysia
UV	–	Ultraviolet
$ZnCl_2$	–	Zinc chloride
ZnO	–	Zinc oxide
Zn^{2+}	–	Zinc ion
$\Delta G°$	–	Gibbs free energy
$\Delta H°$	–	Enthalpy
$\Delta S°$	–	Entropy

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A.1	Calibration graph of rhodamine B (RB) at 575 nm	144
A.2	Textural characteristics & yield of activated carbons	145
B.1	Equilibrium study of ZCBR series (from left to right: control, ZCBR-0.5, ZCBR-1.0, ZCBR-2.0, ZCBR-2.5) at (a) 10 ppm and (b) 50 ppm	146
B.2	Equilibrium study of KCBR series (from left to right: control, KCBR-0.5, KCBR-1.0, KCBR-2.0, KCBR-2.5) at (a) 10 ppm and (b) 50 ppm	146
B.3	Equilibrium study of MCBR-1.0 and FCBR-1.0 (from left to right: control, MCBR-1.0, FCBR-1.0) at (a) 10 ppm and (b) 50 ppm	146
B.4	Equilibrium study of composite series (from left to right: control, KZCBR, MZCBR, FZCBR) at (a) 10 ppm and (b) 50 ppm	147
B.5	Kinetics study of ZCBR-1.0 (from left to right: control, 1 h, 3 h, 6 h, 24 h, 31 h, 48 h, 72 h) at 50 ppm	147
B.6	Regeneration study of spent ZCBR-1.0 (left: irradiated water; right: hot water) after desorption	147

CHAPTER 1

INTRODUCTION

1.1 Research Background

Industrialization is a main key to the economic development, but it is also the root cause of environmental issue. In Malaysia, textile production is not just a fashion trend, but it is also known as the artistic legacy. However, due to the high customer demand, and improper industry effluent management, dye pollution has been a serious threat to the public health and the environment. Therefore, dye wastewater treatment issues must be faced up.

Dye is a visible pollutant. It existence affects not only the quality of surface water, but also changes the aquatic ecosystems as well as reduces the light penetration. Dyes can cause eye burns in humans and animals, methemoglobinemia, cyanosis, convulsions, tachycardia, dyspnoea, irritation to the skin, and if ingested, may lead to irritation to the gastrointestinal tract, nausea, vomiting and diarrhea (Senthikumaar *et al.*, 2005).

There are various treatment methodology have been investigated such as biodegradation, coagulation, oxidation, adsorption and so on. Adsorption is the most economical attractive due to the flexibility and simplicity of design, ease of operation and insensitivity to toxic pollutants (Robinson *et al.*, 2001; Gupta and Suhas, 2009). This process creates a film of adsorbate on the surface of the adsorbent. Besides, when compared with other physico-chemical treatment methods, adsorption is more inexpensive and does not produce sludge (Demirbas *et al.*, 2008). Activated carbon is widely used as adsorbent for dye adsorption due to its large porous surface area, controlled pore structure and inert properties (Walker and Weatherley, 1997).

Activated carbon can be prepared from a variety of raw materials, especially agricultural by-products such as coconut shells, used tea leaves, orange peels and so on (Hu and Srinivasan, 1999; Arami *et al.*, 2005; Tahir *et al.*, 2009). These products are regarded as waste and can caused serious disposal problem in some countries. Therefore, converting them into activated carbon is a feasible solution to the environmental problem. In this study, castor bean residue was used as the precursor of activated carbon.

Malaysia is situated in tropical zone with enough rain and sunlight that suit to castor plant. Castor oil derivatives are similar to petroleum derivatives, thus it is a perfect alternative to petroleum. Furthermore, there is a huge potential for castor oil to be used as biodiesel for vehicle. By year 2015, the global demand for castor oil is estimated to be around 2 million tons. Castor bean residue is the by-product of biodiesel production which remains after the extraction of the oil and comprises about 50% of the weight of castor bean (Robb *et al.*, 1974), which is 1.1 tons per every 1 ton of castor oil production (Santos *et al.*, 2014). Moreover, castor bean residue has no viable application as it contains ricin, a protein that is toxic to cattle (Madeira *et al.*, 2011). Due to its abundant source, castor bean residue is seen as a suitable candidate to replace the conventional precursor of activated carbon.

The preparation of activated carbon via chemical activation involves the use of activating agents such as K_2CO_3, $ZnCl_2$ or KOH. During pyrolysis of cellulose, an organic compound with six carbon ring structure known as levoglucosanis formed, and results in the formation of tar. Some of the pores on carbon are filled or partially blocked by tars and consequently its adsorption capacity becomes lower. Activating agents are functioned as dehydrating agents to inhibit the formation of tar during the pyrolytic decomposition (Derbyshier *et al.*, 1995). Hence, higher yield and better development of porosity are always found in the case of chemical activation when compare to the physical activation. Moreover, lower activation temperature and shorter time are required for chemical activation process (Lim *et al.*, 2010). Zinc chloride ($ZnCl_2$) is a well-known activating agent in the synthesis of activated carbon for wastewater treatment. However, zinc cation is a well-known pollutant in aqueous solution. It is toxic to the aquatic organism and may cause long-term adverse effects to the aquatic environment. So, less hazardous metals chloride salts were investigated in this study to replace $ZnCl_2$ as activating agent.

1.2 Problem Statement

$ZnCl_2$ is a widely used activating agent in the preparation of activated carbons for research, but there are concerns about the aquatic toxicity of $ZnCl_2$ in large-scale manufacturing process. Because it is a powerful Lewis acid, zinc cation in aqueous solution gives corrosive effect to bacteria, plants, invertebrates and vertebrate fish. Zinc toxicity may take months to resolve because there is no particular body store for zinc when it dissolves in HCl in stomach (Nriagu, 2007). Less toxic metals chloride such as KCl, $MgCl_2$ and $FeCl_3$ have similar characteristics to $ZnCl_2$ in aqueous solution, which opens up the possibility of replacing $ZnCl_2$ as activating agent in the preparation of activated carbon (Rufford *et al.*, 2010).

1.3 Objective

Three objectives in this research are stated below:

i. To synthesize and characterize activated carbons from castor bean residue by different metals chloride salts activation.
ii. To establish the adsorptive studies of rhodamine B by activated carbons at different initial concentrations, time intervals and temperatures.
iii. To evaluate the regeneration of spent-activated carbon using hot water and irradiated water for three consecutive cycles.

1.4 Scope of Study

i) To synthesize and characterize activated carbons prepared from castor bean residue by different metals chloride salts activation.

Impregnation was carried out using metals chloride salts as activating agents, which are $ZnCl_2$, KCl, $MgCl_2$, $FeCl_3$ and composites of metal chloride salts at various impregnation ratios from 0.5 to 2.5. Heating temperature and heating period were fixed at 550°C and 1.5 h, respectively. Activated carbons were characterized based on specific surface area, morphology, surface functional group, Boehm titration and elemental analysis.

ii) To establish the adsorptive studies of rhodamine B by activated carbons at different initial concentrations, time intervals and temperatures.

The studies were carried out for the best-performed activated carbons from each activation series. Rhodamine B was used as adsorbate model. Four isotherm models which are Langmuir, Freundlich, Redlich-Peterson and Dubinin-Radushkevich were used to fit the adsorption data at different initial concentrations. Rate of adsorption for three initial concentrations at different time intervals 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, $\triangle G^o$, enthalpy, $\triangle H^o$ and entropy, $\triangle S^o$ were investigated through the effect of temperature on dye adsorption from 20 to 55°C for the best-performed activated carbon.

iii) Regeneration of spent-activated carbon using hot water and irradiated water.

The regeneration study was performed using hot water and irradiated water for three consecutive adsorption-desorption cycles to determine the regeneration efficiency and recovery of activated carbon.

1.5 Significance of Study

This study is carried out to give further understanding on the contribution of agricultural waste activated carbon to dye-containing wastewater treatment. The issues related to castor bean waste management can be reduced if it is converted into activated carbon. Less-toxic activating agents are investigated as an alternative to $ZnCl_2$ in the preparation of activated carbon for dye removal. This study also proposes an environmental friendly approach for regeneration method of spent activated carbon.

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