REMOVAL OF REACTIVE BLACK 5 BY ACTIVATED CARBON FROM PINEAPPLE LEAVES VIA ULTRASONIC ASSISTED ADSORPTION

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DEDICATION

To my beloved parents,

Abd Latif Jusoh
Noraini Rahim

To my supervisor,
Assoc. Prof. Ir. Dr. Norzita Binti Ngadi

To each of you

And

To myself

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All the praises belong to Allah SWT

The road to success has never easy to navigate but the time you get to read this on the printed thesis, I made it! The least I could do is expressing my greatest gratitude to each of you I crossed path with throughout this amazing research journey.

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ABSTRACT

Adsorption is a prominent process for the treatment of dye from textile wastewater due to its simplicity. However, usage of commercial activated carbon in adsorption is costly and inconvenient for regeneration process. Hence, there is a need to shift towards new adsorbents that are low cost and highly efficient. Therefore, this study focused on the synthesis of new activated carbon with pineapple leaves as a precursor for the removal of reactive black 5 (RB5) dye. Chemical activation method was employed to tailor the internal porous structure and surface area of the adsorbent. Treatment with sodium hydroxide had achieved high removal compared to other activating agents. The effect of four preparation variables: impregnation ratio, impregnation time, carbonization temperature and carbonization time on RB5 uptake from aqueous solution were investigated. Based on the Box-Behnken design, a quadratic model was developed to correlate the preparation variables to the RB5 uptake. The significant factor in each experimental design response was identified from the analysis of variances. The optimum pineapple leaves activated carbon (PLAC) was obtained using an impregnation ratio of 3.75, carbonization temperature of 517°C and carbonization time of 1 hour 22 minutes. Then, the synthesized PLAC was characterized using field emission electron microscopy, Fourier transform infrared spectroscopy, Brunauer Emmett Teller surface area, energy dispersive X-ray, and point of zero charge. A batch experiment was conducted under ultrasound-assisted adsorption. The results obtained were excellent with almost up to 100% RB5 removal under the following conditions; 35 min of contact time, 0.1 g of adsorbent, and 0.05 g/L of initial RB5 concentration, pH 5 and at 30 °C. Kinetic, isotherm and thermodynamics evaluation were also performed for the adsorption data. The adsorption data fitted well to the pseudo-first-order model with the influence of intraparticle diffusion. For the isotherm, the data best fitted to the Langmuir model with the maximum adsorption capacity of 103 mg/g. A thermodynamics analysis showed that the adsorption was exothermic and spontaneous. The PLAC can be reused five times with the percentage removal above 60%.

ABSTRAK

Penjerapan adalah proses penting untuk penyingkiran pencelup daripada sisa air buangan teskstil kerana kesederhanaannya. Walau bagaimanapun, penggunaan karbon aktif komersial dalam penjerapan adalah mahal dan sukar bagi proses penggunaan semula. Oleh yang demikian, terdapat keperluan untuk beralih kepada penjerap baharu berkos rendah dan juga berkesan. Maka, kajian ini memfokuskan pada sintesis karbon aktif baharu dari daun nanas untuk penyingkiran pencelup reaktif hitam 5 (RB5). Kaedah pengaktifan kimia telah digunakan untuk mengubahsuai struktur dan permukaan kawasan berliang dalam penjerap. Proses rawatan dengan natrium hidroksida mencapai penyingkiran yang tinggi berbanding dengan agen pengaktif lain. Kesan empat pemboleh ubah penyediaan: nisbah impregnasi, masa impregnasi, suhu karbonisasi dan masa karbonisasi terhadap penyingkiran RB5 dari air telah dikaji. Berdasarkan reka bentuk Box-Behnken, model kuadratik telah digunakan untuk mengkaji hubungan antara pemboleh ubah penyediaan karbon aktif terhadap pengambilan RB5. Faktor penting dalam setiap tindak balas reka bentuk eksperimen dikenal pasti dari analisis varian. Karbon aktif daun nanas (PLAC) optimum dapat diperolehi dengan menggunakan nisbah impregnasi 3.75, suhu karbonisasi 517 °C dan masa karbonisasi 1 jam 22 minit. Kemudian, PLAC yang disintesis dikaji ciri cirinya dengan menggunakan mikroskop elektron imbasan pancaran medan, spektroskopi inframerah jelmaan Fourier, analisis luas permukaan Brunauer Emmett Teller, penyebaran tenaga sinar-x, dan titik cas sifar. Kajian penjerapan secara kelompok telah dijalankan di bawah penjerapan berbantu ultrabunyi. Hasil yang diperoleh amat memuaskan dengan penyingkiran RB5 hampir 100% pada keadaan berikut; 35 minit masa sentuh, 0.1 g dos penjerap, dan 0.05 g/L kepekatan awal RB5, pH 5 dan pada 30 °C. Penilaian kinetik, isoterma dan termodinamik juga dilakukan untuk data penjerapan. Data penjerapan sesuai dengan model kinetik pseudo-tertib-pertama dengan pengaruh penyebaran intrapartikel. Bagi kajian isoterm, didapati data paling sesuai dengan model Langmuir dengan kapasiti penjerapan maksimum sebanyak 103 mg/g. Analisis termodinamik menunjukkan bahawa penjerapan adalah bersifat eksotermik dan spontan. PLAC dapat digunapakai semula sebanyak lima kali dengan peratusan penyingkiran pencelup melebihi 60%.

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LIST OF ABBREVIATIONS

AC	-	Activated Carbon
BBD	-	Box-Behnken Design
BET	-	Brunauer-Emmet-Teller
BOD	-	Biochemical Oxygen Demand
C	-	Carbon
C.I	-	Color Index
$CaCl_2$	-	Calcium Chloride
CCD	-	Central Composite Design
COD	-	Chemical Oxygen Demand
EDX	-	Energy Dispersive X-ray
FAO	-	Food and Agricultural Organization
FeCl	-	Iron Chloride
$FeSO_4$	-	Iron Sulphate
FFD	-	Full Factorial Design
FTIR	-	Fourier Transform Infrared Spectroscopy
H	-	Hydrogen
H_2SO	-	Sulphuric acid
H_3PO_4	-	Phosphoric acid
HCl	-	Hydrochloric acid
IUPAC	-	International Union of Pure and Applied Chemistry
KOH	-	Potassium Hydroxide
MV	-	Methyl Violet
MO	-	Methyl Orange
$MgCl_2$	-	Magnesium Chlroide
N	-	Nitrogen
NaOH	-	Sodium Hydroxide
O	-	Oxygen
OFAT	-	One-Factor-at-A-Time
pH	-	Potential of Hydrogen
pH_{PZC}	-	Point of zero charge

PL - Pineapple Leave
PLAC - Pineapple Leave Activated Carbon
PFO - Pseudo First Order
PSO - Pseudo Second Order
RB5 - Reactive Black 5
RSM - Response Surface Methodology
S - Sulphur
SEM - Scanning Electron Microscope
SME - Small Medium Enterprise
UV - Ultraviolet
$ZnCl_2$ - Zinc Chloride

LIST OF SYMBOLS

Å - Angstrom
Co - Initial concentration of parameters
Cf - Final concentration of parameters
Ce - Concentration at equilibrium
°C - Degree Celcius
% - Percentage
g - gram
K_F - Freundlich constant
K_L - Langmuir constant
m - mass
mg/g - milligram per gram
mg/L - milligram per litre
ml - millilitre
q_e - Equilibrium adsorption capacity
R^2 - Correlation coefficient
rpm - Rotation per minute
wt% - Weight percent
V - Weight percent
v/v - volume over volume

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Batik industries are among rapidly growing textile industries in Malaysia, particularly in East Coast States Malaysia such as Kelantan and Terengganu. This industry has positively generated enormous contribution to Malaysia economic growth due to high demands locally and internationally [1, 2]. Despite its contribution in batik industries, there is a major concern about a large volume of the dye-bearing effluent discharge from *batik* processing including their negative impact to the environment and aquatic life even at low concentration [3].

There are various types of dyes commonly used in *batik* industries such as reactive dye. This dye is the brightest class of soluble dyes and has a complex molecular structure that could make them more stable and remain in the environment for an extended period [4]. Among reactive dyes, Reactive Black 5 (RB5), which also represents an anionic group, has gained interest among researchers due to their hazardousness in the environment. According to Gottlieb *et al.* [5], the allowable concentration of anionic RB5 for humans is 27.5mg/L, which depicts that excessive use of RB5 could cause a severe effect to the environment and human health due to mutagenic and carcinogenic properties. Therefore, a proper and effective technology for dye wastewater treatment is required before discharged to the environment. Additionally, stringent regulation imposed by the government on the green industry has given more pressure to the textile industry as they require to design effective standard methods for their effluents [6].

Many technologies have been introduced to treat *Batik* effluent, such as chemical, biological, and physical [7]. However, chemical and biological process present a few drawbacks such as complex operation, high amount of chemical usage and high sludge generation. Furthermore, RB5 used in *batik* processing are brightly coloured, water-soluble, highly reactive and toxic [8] which are detrimental to microorganism activity consequently inefficient for biological methods. Meanwhile, a physical approach such as adsorption by activated carbon (AC) offers a simple design, ease of operation, and insensitivity towards toxic substance, bringing more advantages than chemicals and biological. In this regards, this approach has become the focus in recent studies of dye effluent decolourization generated from *batik* manufacturing. It is worth mentioning that the efficiency of activated carbon as an adsorbent depends on its pore size, surface area and mesoporous structure. Therefore, the development of carbon materials with controlled porosity is crucial to achieving high adsorption capacity.

Additionally, there is growing interest in using low-cost material for the adsorption of pollutants. For instance, Wong *et al.* [9] has successfully synthesized AC derived from spent tea leaves to remove aspirin from wastewater with an adsorption capacity of 178.57 mg/g. Another study was reported by Nasrullah *et al.* [10] on the synthesis of AC from mangosteen peel to remove methylene blue with an adsorption capacity of 1193 mg/g. Among all, utilization of agricultural waste or by-product as AC gives great merit because of its abundance and consists of cellulose and lignin components that are feasible for adsorption [11, 12]. Pineapple leaves (PL) which primarily cultivated in tropical countries especially Asia, consist of cellulose (70-82%), lignin (5-12), and hemicellulose, which provide the basis for its potential to use as an economical adsorbent [13, 14].

Thus, this research aims to determine the efficiency of the physical method by adsorption with AC derived from agricultural waste PL for synthetic dye treatment, RB5.

1.2 Problem Statement

Adsorption by AC is most widely in wastewater treatment due to its effectiveness and high adsorption capacity to adsorb large quantities of contaminants, including dyes. However, AC's application is often restricted due to the high cost associated with the starting materials and extensive energy consumption and chemicals required for synthesis on a large scale of wastewater treatment. To address the aforementioned issue, current research interest lies in developing less complex AC from cheap sources, such as agricultural waste that is low cost, easy access and abundant in nature.

Several studies demonstrated satisfactory performance of dye removal using agricultural waste such as walnut shell, coconut husk, bamboo, cassava peel, etc. However, up to date, little attention is given to PL explored as eco-friendly and low-cost adsorbent. Pineapple (*Ananas comosus*) is a herbaceous perennial plant that belongs to one of the world's essential commercial tropical fruit crops, especially in the Asia region. Due to the massive production of pineapple plants, there is an abundance of residues and by-products generated from the plants (such as its leaves), which resulted in secondary pollution and environmental problem. In this regard, it is of great merit to study the potential of PL as a useful AC in removing pollutant RB5 from wastewater. Conversion of PL to AC adds values to such agricultural waste, which otherwise requires extra costs for disposal.

Although adsorption by AC from agricultural waste materials has widely been accepted due to its effectiveness and low cost, the adsorption process involving ultrasonic waves is of great advantage. It helps increase the mass transfer between adsorbent and adsorbate. Thus, less time needed and adsorption efficiency is improved. Hybrid systems involving ultrasonic-assisted method has not been widely explored well before in the field of environment. Ultrasound irradiation is regarded as a motivation force in a chemical process to accelerate mass transfer due to the acoustic cavitation phenomenon with its formation, growth and collapse of micrometrical bubbles.

Ultrasound has been proven to be a handy tool in intensifying the mass transfer between the active adsorption sites of the adsorbent process and breaking the affinity between adsorbate and adsorbent. In this way, the ultrasonic waves can enhance the current conventional agitation adsorption and increase the kinetic.

Therefore, this study aimed to look at the potential and efficiency of utilization of abundance waste from the pineapple industry that is PL as AC for the removal of RB5 in the presence of ultrasound.

1.3 Research Objective

This study aims to achieve the following objectives:

1. To optimize the preparation condition of PLAC using the chemical activation method for the removal of RB5 and to characterize the PLAC.
2. To investigate the effect of adsorption process parameters on the performance of PLAC for RB5 removal during the ultrasonic-assisted adsorption process.
3. To identify the adsorption kinetics, isotherm and thermodynamic of the PLAC for the removal of RB5.

1.4 Research Scope

This research was extended into a more technical scope. The PLAC was synthesized via a chemical activation method. The porosity of the activated carbon was modified by varying the activating agent (zinc chloride; $ZnCl_2$, phosphoric acid; H_3PO_4, potassium hydroxide; KOH and sodium hydroxide; NaOH), impregnation ratio of raw material and agent (0.25 to 4) and time (1 to 24 hour), carbonization temperature (300 to 700°C) and time (1 to 5 hour).

This condition for preparation activated carbon was optimized via Response Surface Methodology (RSM) using Design- Expert ® software (version 9, Stat-Ease, Inc., Minneapolis, USA) to determine optimum condition interaction between variables.

The optimum synthesized activated carbon was selected for characterization that is BET and FESEM for porosity and surface morphology, respectively. EDX, FTIR, and pHpzc also have been further characterized.

Adsorption from RB5 solutions was carried out, where parameter that was analyzed during adsorption process are; a) effect of contact time (5 to equilibrium), b) effect of initial dye concentration (50 to 250 mg/L), c) effect of pH (3 to 12), d) effect of adsorbent dosage (0.05 to 1g) and effect of temperature (30 to 80 °C).

The dyes adsorption mechanism onto the PLAC were determined via adsorption isotherm using Langmuir and Freundlich. The adsorption kinetics were also presented in this investigation by pseudo-first-order, pseudo-second-order and intraparticle diffusion model. In addition, thermodynamics evaluation was also conducted.

Reusability study was done on PLAC by simple washing process with distilled water throughout five cycles, and the removal of RB5 was calculated for each cycle.

1.5 Significance of Research

This study offers an alternative to commercial activated carbon as it is prepared from easy access to waste materials and renewable (PL). Conversion of pineapple residue into useful adsorbent provides an alternative way to reduce waste and minimise disposal costs. Treating this residue to a beneficial product can minimize the cost of disposal and conquer environmental pollution.

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