REMOVAL OF REACTIVE BLACK 5 DYE USING MODIFIED CHITOSAN-PANDAN ADSORBENT

FATIN AMIRAH RAZMI

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To my beloved father and mother,

Razmi Mohd Yunos

Masrinah Md Nor

To my supervisor,

Assoc. Prof. Dr. Norzita Ngadi

Also to all my friends,

Thank you for your love, support and guidance.

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ABSTRACT

There has been increased interest of chitosan as a dye adsorbent. Nonetheless, the chitosan tend to form bonding between their monomer chains that result in a rigid structure which affects its capability for dye adsorption. This is why there are many recent studies on modified chitosan through various modification in order to overcome the limitation. But, most modifications involve chemical additive agents as well as complex procedures that are conducted under strong conditions. Not much research consider low-cost organic materials as modifying agents. The purpose of this work is to investigate the performance of chitosan modified with *pandan* leaves in the adsorption of reactive black 5 (RB5). Modified chitosan-pandan (MCP) were synthesised by simple wet impregnation using polyphenols from extracted *pandan* oil under mild condition. MCP was characterised using the scanning electron microscopy, Fourier transform infrared spectroscopy and Brunauer-Emmett-Teller surface area analysis. A batch adsorption was conducted to study the effect of retention time (0-40 min), initial dye concentration (100-1000 mg/L), pH (3-11), temperature (25-80 °C) and dosage of adsorbent (0.01-1.0 g), and to determine the optimum process conditions. Kinetics, isotherm and thermodynamics evaluation were also performed on the adsorption data. The results of MCP adsorption showed outstanding dye removal, with almost 100% under the optimum conditions (30 min, 200 mg/L of RB5, pH 7 and 0.1 g of MCP). The adsorption data fitted well to the pseudo-second order model, indicating the role of chemisorption with the influence of intraparticle diffusion. For isotherm study, the data are best fitted to the Langmuir model (R^2 = 0.95) with the maximum adsorption of 115.58 mg/g. A thermodynamics analysis showed that the adsorption was endothermic, occurred spontaneously and feasible. MCP is capable to be regenerated up to 5 times with percentage removal above 50% by only washing with distilled water. In conclusion, a satisfactory performance of MCP in RB5 removal was successfully demonstrated, can be used as a new promising adsorbent for the removal of dyes from textile wastewater.

ABSTRAK

Terdapat peningkatan minat terhadap kitosan sebagai penjerap pencelup. Namun, kitosan cenderung untuk menghasilkan ikatan sesame rantaian monomer dan membentuk struktur kukuh yang mana mempengaruhi keupayaannya untuk menjerap. Oleh itu, pelbagai kajian terhadap kitosan terubahsuai melalui pelbagai pengubahsuaian bagi mengatasi kelemahan ini. Tetapi, kebanyakan pengubahsuaian melibatkan agen bahan additif kimia serta prosedur yang kompleks dilaksanakan dalam keadaan yang melampau. Hanya segelintir kajian sahaja yang menggunakan bahan organik murah sebagai agen pengubahsuaian. Tujuan kajian ini adalah untuk mengkaji prestasi kitosan terubahsuai dengan daun pandan untuk penjerapan pencelup hitam 5 (RB5). Terubahsuai kitosan-pandan (MCP) telah dihasilkan melalui rawatan basah antara kitosan dan polifenol yang diperolehi daripada ekstrak minyak pandan. MCP dicirikan menggunakan mikroskop elektron imbasan, spektroskopi intra merah jelmaan Fourier dan analisis luas permukaan Brunauer-Emmett-Teller. Penjerapan kelompok dijalankan untuk mengkaji kesan terhadap masa simpanan (0-40 min), kepekatan awal pencelup (100-1000 mg/L), pH (3-11), suhu (25-80 °C) dan dos penjerap (0.01-1.0 g), bagi mengenalpasti keadaan proses optimum. Penilaian kinetik, isoterma dan termodinamik juga dijalankan menggunakan data penjerapan. MCP telah menunjukkan penjerapan RB5 yang baik iaitu hampir 100% pada keadaan optimum (30 min, 200 mg/L RB5, pH 7 dan 0.1 g MCP). Model pseudo-tertib kedua adalah yang paling sesuai dengan data penjerapan, menunjukkan berlakunya penjerapan kimia di bawah pengaruh resapan antara zarah. Untuk kajian isoterma, data membuktikan model Langmuir adalah yang paling sesuai (R^2= 0.95) di mana penjerapan maksimum ialah 115.58 mg/g. Analisis termodinamik menunjukkan bahawa penjerapan adalah endotermik, berlaku secara spontan dan boleh dilaksanakan. MCP juga boleh digunapakai sebanyak 5 kali dengan peratus penyingkiran melebihi 50% hanya melalui proses cucian menggunakan air suling. Kesimpulannya, penjerap MCP menunjukkan prestasi yang memuaskan dan boleh digunakan sebagai penjerap baharu yang berpotensi untuk penyingkiran pencelup dari sisa air buangan tekstil.

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LIST OF ABBREVIATIONS

USA	-	United State America
IUPAC	-	International Union of Pure and Applied Chemistry
MCP	-	modified chitosan-pandan
RB5	-	reactive black 5
GCMS	-	Gas chromatography combined with mass spectroscopy
FTIR	-	Fourier transform infrared spectroscopy
SEM	-	scanning electron microscopy
BET	-	Brunauer-Emmett-Teller
BJH	-	Barret-Joyner-Halenda
UV-Vis	-	Ultraviolet-visible
D-R	-	Dubinin-Radushkevich
n.a.	-	not available

LIST OF SYMBOLS

λ_{max}	-	maximum wavelength (nm)
R_1-N=N-R_2	-	azo group
P/P_o	-	relative pressure
HCI	-	hydrochloric acid
NaOH	-	sodium hydroxide
N_2	-	nitrogen gas
BAH	-	Butanoic acid, 3-hydroxy-
GHMP	-	4-Geranyloxy-3-hydroxy-5-methoxyphthalaldehyde
pH	-	potential of hydrogen
W_w	-	wet sample weight (g)
W_D	-	weight sample after drying (g)
q	-	amount of adsorption capacity of dye onto adsorbent (mg/g)
$q_{e,exp}$	-	amount capacity of dye onto adsorbent at equilibrium obtained from the experimental data (mg/g)
$q_{e,cal}$	-	amount capacity of dye onto adsorbent obtained by calculating from the model (mg/g)
$q_{e, mean}$	-	mean of $q_{e,exp}$ values (mg/g)
q_e	-	equilibrium adsorption capacity (mg/g)
q_t	-	amount of adsorption at time t (mg/g)
C_o	-	initial concentration (mg/L)
C_f	-	final concentration (mg/L)
R^2	-	coefficient of determination
χ^2	-	chi-squared
m	-	weighed adsorbent (g)
V	-	volume of solution (L)
k_1	-	rate constant for the pseudo first order adsorption (min^{-1})

k_2	-	rate constant of the pseudo second order adsorption (g/mg min)
k_p	-	intraparticle diffusion constant (mg/g min)
t	-	time (min)
C	-	reflects of boundary layer effect (mg/g)
C_e	-	dye concentration in solution at equilibrium (mg/L)
q_{max}	-	maximum adsorption capacity (mg/g)
b	-	Langmuir constants (L/mg)
R_L	-	separation factor
K_f	-	adsorption capacity (mg/g)
n	-	indicated of favourability
K_T	-	equilibrium binding constant or maximum binding energy (L/mg)
B	-	heat of adsorption (J/mol)
β	-	Temkin constant
T	-	absolute temperature (K)
R	-	universal gas constant (J/mol K)
Q_m	-	theoretical adsorption capacity (mg/g)
k_e	-	constant related to adsorption energy (mol^2/kJ)
ε	-	Polanyi potential
E	-	mean free energy of adsorption (J/mol)
ΔH	-	enthalpy change (kJ/mol)
ΔS	-	entropy change (kJ/mol K)
ΔG	-	Gibb's change (kJ/mol)
k_d	-	standard thermodynamics equilibrium constant
%	-	percentage
°C	-	Celsius
K	-	Kelvin
min	-	minute
min^{-1}	-	inverse minute
g	-	gram
mg	-	miligram
L	-	liter
mL	-	mililiter

g/L	-	gram per liter
mg/L	-	milligram per liter
mg/g	-	milligram per gram
kV	-	kilovolt
cm	-	centimeter
cm^{-1}	-	inverse centimeter
M	-	molarity (mol/L)
nm	-	nanometer
μm	-	micrometer
rpm	-	revolution per minute

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Colour plays an important role in everyday life due to its extensive use for clothing, equipment, plastic, paper, cosmetic, medicine, food and beverage (Kumar and Jiang, 2016). Many manufacturers compete to produce synthetic dyes at a lower cost, while having brilliant colours and high absorptivity onto surface of various materials (Ngah *et al.*, 2011). Therefore, the demand of dyes in the market has increased significantly for the past decades. According to Zhang *et al.* (2015), over 7×10^5 tons dyes are expected to have been manufactured per year. As shown in Figure 1.1, Asia is the main producer of dyes in the world.

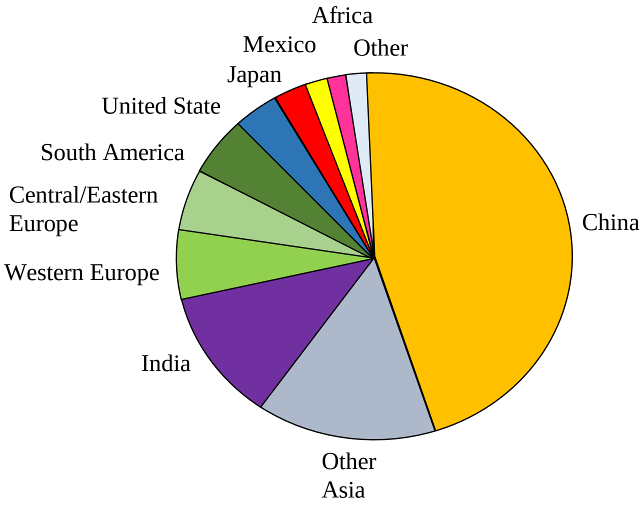

Figure 1.1 World consumption of synthetic dyes (Chemical Economic Handbook, 2014)

Following the development of dye industry in Malaysia, environmentalists have voiced their concern over the fate of wastewater produced by dye manufacturing plants. In Malaysia, Pang and Abdullah (2013) reported that the amount of the production of dyes wastewater has increased from the year 2007 (744 tons) to 2009 (1559 tons) and the number is expected to rise each year due to the increased demand of synthetic dyes in the market.

Among all dyes, azo (R_1-N=N-R_2) constituents are considered as the largest chemical class of dyes used in textile industries, and they consist about 60-70 % of all dyes produced worldwide (Popli and Patel, 2015). There are more than 2,000 applications of azo dyes in many industries that are mostly toxic to the environment and humans (Tahir *et al.*, 2016). The reactive black 5 (RB5) is one of azo dyes that is frequently used in the apparel industry such as cotton, wool, nylon and synthetic fibres (El Bouraie and El Din, 2016). This is due to the properties of RB5 that are easy to use, cost-effective and offers brilliant colours.

Azo group of RB5 dyes is basically connected to benzene whereas under a reduction condition, it can be chemically broken down to aromatic amine that is considered as toxic, carcinogenenic, tetragenic and mutagenic compounds (Chequer *et al.*, 2015). Moreover, RB5 dyes are structurally complex that are chemically stable, persistent and non-biodegradable (Vakili *et al.*, 2014). There is a work by Zainudin *et al.* (2016) that tested the chemical stability of RB5 dyes and found that it has high solubility and a strong resistance to changes of acids, alkalis, heat and light. Thus, it is a great challenge to remove RB5 dyes from wastewater.

Furthermore, the presence of dyes in water bodies, such as lakes and river, hinder the penetration of sunlight into the water. Aquatic plants need sunlight for oxygen production that helps to protect and sustain aquatic biodiversity (Hassan, 2015). Besides that, the intermediate compounds generated by azo dyes can threaten aquatic life. Azo dyes also jeopardize the health of humans, as the dyes can easily be absorbed into the skin pores and accumulate in human body (Sudha *et al.*, 2014). Under long-term exposure, human bodies will suffer adverse health risks such as compromise the functions of liver, kidneys, sexual organs, as well as brain and nervous system (Yagub *et al.*, 2014). In short, uncontrolled discharge of dye wastewater from municipalities and industries into the water body puts the environment, aquatic life and humans at high risk. Thus, it is important to determine the suitable treatment for dye wastewater before irreversible damage to the environment and human is done.

There are several advanced techniques that can remove dyes in wastewater, for instance, chemical precipitation (Cao *et al.*, 2014), membrane filtration (Karim *et al.*, 2014), electrochemical advanced oxidation (Mansur *et al.*, 2014) and coagulation (Wang *et al.*, 2017). However, these methods exhibit the limitations of relatively high capital and operational costs due to chemicals and energy input of the processes. Some of the methods, which is advanced oxidation, may produce a potentially hazardous by-product due to formation of oxidation intermediate compounds. On the other hand, adsorption is recognised as an attractive and versatile method used in current developed countries, due to its simplicity in operation, as well as low material and operational costs in removing dyes in wastewater (Mohammed *et al.*, 2014). The adsorption method has been extensively used for removal of different organic and inorganic dyes from wastewater. Hence, researchers have intensified investigation efforts into adsorption technology as a solution for dye removal from wastewater.

Commercial activated carbon is an important element in the remediation of wastewater by adsorption, due to the remarkably high effectiveness of removal of pollutants from wastewater. Nevertheless, the high cost of activated carbon continues to limit the expansion application of the process in wastewater treatment. The high production cost is related to the use of chemical reagents as well as high temperature during carbonisation and activation steps. Thus, many studies focusing to utilise various potential natural materials for replacement of commercial activated carbon as an adsorbent, such as clay, zeolites, chitosan, siliceous materials, fly ash, agro-wastes and industrial wastes (Talwar *et al.*, 2016).

1.2 Problem Statement

In recent years, chitosan has received great attention from researchers as a potential adsorbent that could reduce numerous types of dye in wastewater (Liu *et al.*, 2015). The reason is that chitosan has high affinity for most dyes, and is a chemically stable chelating agent with high reactivity (Gul *et al.*, 2016). Furthermore, chitosan is known as the second most abundant bio-polymer on earth and originated from crustacean waste. Its non-toxic nature combined with high biodegradability and biocompatibility makes it as highly potential adsorbent in wastewater treatment.

Despite the beneficial properties, chitosan has poor solubility in water due to intermolecular and intramolecular of hydrogen bonding interaction between their polymer chains. This property, along with its low mechanical strength and poor acid resistance causes serious challenges in application of wastewater treatment. Thus, there is a great number of reports on the modification of chitosan in many ways especially in terms of chemical modification due to the existence of amino and hydroxyl groups in its molecular structure (Kyzas and Bikiaris, 2015).

However, most of the modifications are complex, include the addition of synthetic chemicals and require strong acidic or base reagents along with the process preparation. The possible undesirable reactions in the chemical mixtures may produce hazardous by-products. Therefore, there is a need to investigate potential natural additive agents for chitosan modification. It is also necessary to modify chitosan without using any synthetic additive by diverting to low-cost organic material such as *pandan* leaves. The objective of this study is to investigate the property of modified chitosan-pandan (MCP) synthesised in mild condition via simple wet impregnation as a dye adsorbent.

Pandan as reported by Ningrum and Schreiner (2014), contains a high amount of polyphenol acids (gallic acid, ferulic acid and cinamic acid), flavonoids (catechin) and amino acids (glutamic acid, aspartic acid, threonine, serine, histidine, alanine, and proline). These natural polyphenols are responsible for breaking down the intermolecular and intramolecular of hydrogen bonds between chitosan monomers (Hu and Luo, 2016). It was also reported that natural polyphenols have a relatively high capacity of antioxidant and the ability to scavenge free radicals (Dai and Mumper, 2010). The antioxidants also appear to be a good alternative modifying agent that helps to increase the hydroxyl group towards the backbone of chitosan (Hu and Luo, 2016). Therefore, natural polyphenols from *pandan* leaves could increase the adsorption capacity by improving physicochemical properties of chitosan. Furthermore, there has been no study reported on the modification of chitosan with *pandan* leaves as a dye adsorbent.

1.3 Objective of the Study

The objectives of this study are identified below:

i. To synthesise and characterise modified chitosan-pandan (MCP) as dye adsorbent.

ii. To investigate the adsorption performance of MCP.

iii. To analyse the adsorption of kinetics, isotherm and thermodynamics of MCP.

1.4 Scope of the Study

This study is limited to the following scopes:

i. The modification of chitosan was carried out by simple wet impregnation method using natural polyphenol from extracted *pandan* oil. The extracted *pandan* oil was obtained by maceration technique using 95 % ethanol as a solvent.

ii. Screening test, seven MCP adsorbents were synthesised with different amount of chitosan (0.01-1.00 g) dissolved in 50 mL of extracted *pandan* oil in order to determine the best efficient removal of RB5 in aqueous solution.

iii. The MCP was characterised in terms of surface morphology, functional groups and surface area using the scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET) respectively.

iv. The optimal adsorption condition of MCP was obtained from the batch adsorption analysis with the following range for each parameter; retention time (0 to 40 min), initial dye concentration (100 to 1000 mg/L), initial pH solution (3 to 11), reaction temperature (25 to 80 °C) and dosage of adsorbent (0.01 to 1.0 g). The regeneration of study of MCP was also evaluated. The response for this study was percentage removal of RB5.

v. The models used for the analysis include: pseudo first-order, pseudo second-order model and intraparticle diffusion model (for kinetics assessment), Langmuir, Freundlich, Temkin and Dubinin-Radushkevich model (for isotherm analysis). For thermodynamics evaluation, the process spontaneity, changes in process enthalpy and entropy were identified based on the adsorption data.

vi. The seven consecutive runs for regeneration of MCP by washing using distilled water.

1.5 Significance of the Study

As discussed previously, most of the chitosan modifications are complex, use chemical additive agents and require strong acidic or base reagents along with the process preparation. Thus, this research proposed a greener approach of modification of chitosan without using synthetic additive agent, instead of using a low-cost organic material that is *pandan* leaves. *Pandan* leaves have many benefits. It is cheap, renewable resource, abundant, safe and biodegradable.

Therefore, by using *pandan* leaves as a modifying agent for chitosan there are many advantage that can be related to such modification. One of it is that the adsorbent is environmental-friendly, as both chitosan and *pandan* leaf are derive from natural sources. Hence, this combination of materials as an adsorbent does not generate any harmful by-product towards the environment. Moreover, in this study, the process modification of chitosan is simple and conducted under mild condition. In addition, there is a possibility that the MCP adsorbent can be useful in reducing odour from wastewater due to the fragrant ester compounds attained from *pandan* leaves (Yanti *et al.*, 2014).

This study contributes a new knowledge for future studies in terms of the ideal natural additive agent that has good properties and high adsorption capacity to modified chitosan. Based on general findings, this study has the potential to give a major contribution to science especially to generate a powerful adsorbent for textile wastewater treatment.

1.6 Thesis Outline

This thesis comprises of five chapters as described below.

Chapter 1 briefly present the introduction, problem statement, objectives, scopes and significance of the study.

Chapter 2 consists of an extensive literature review on chitosan modification methods by other studies, as well as modification of chitosan important aspect of the theory, polyphenol as the grafting agent and *pandan* as the raw material used in this study. Apart from that, the adsorption of previous studies on chitosan modification was also summarised.

Chapter 3 provides detailed description on materials and methods used in the study, including extraction of polyphenols from fresh *pandan*, preparation of MCP adsorbent and screening measurement, characterisation of extracted *pandan*, raw chitosan and MCP. The experimental methods used to study the adsorption performance onto MCP under the influence of retention time, initial dye concentration, pH solution, temperature and dosage of adsorbent are also described together with underlying theories and principles in analysis of adsorption kinetics, isotherm and thermodynamics.

Chapter 4 presents the results obtained from this study, including comparison of raw chitosan and MCP in term of their characterisation results, as well as adsorption performance on RB5. The analysis result acquired from the adsorption reaction, kinetics, isotherm, thermodynamics, regeneration of MCP and finally the comparative results of MCP are also discussed. The corresponding discussion and justification of the results have also been made accordingly.

Chapter 5 concludes the major findings in this study. The recommendations for future research were also discussed in this chapter to improve the structure of the study as well as the findings.

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