UNIVERSITI PUTRA MALAYSIA

REMOVING REACTIVE AND ACID DYES FROM SINGLE AND BINARY SOLUTIONS BY ADSORPTION ON QUATERNIZED KENAF CORE FIBERS

INTIDHAR JABIR IDAN AL-THARWANI

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By

INTIDHAR JABIR IDAN AL-THARWANI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

August 2017

DEDICATION

This thesis is dedicated to my family with all my love

Intidhar Jabir Idan
August 2017

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Doctor of Philosophy

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August 2017

Chairman : Professor Luqman Chuah Abdullah, PhD
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It is estimated that every year 280,000 tonnes of textile dyes are released in textile mill effluent, and unfortunately, all factories are still using water streams for discharging their effluent water. In the wastewater treatment plant, activated carbon is a widely used adsorbent to remove many types of dyes, but it is very expensive. Therefore, adsorption by utilizing different types of agro-residues is one of the alternative materials to remove various types of dyes from solutions.

In the present research, kenaf core fiber (KCF) residue was chemically modified with (3-chloro-2-hydroxypropyl) trimethylammonium chloride to alter the surface properties to capture anionic dyes from aqueous solution. Batch adsorption studies were conducted to investigate the performance of quartenized kenaf core fiber (QKCF) to remove Reactive Red-RB, Reactive Black-5, Acid Blue-25, and Acid Green-25 from a single system. Various parameters such as initial dye concentration, adsorbent dosage, agitation speed, contact time, temperature and pH were investigated for the single solution system.

The present research explored the suitability of quaternized kenaf core fiber (QKCF) to serve as an adsorbent for the removal of anionic dyes from a binary system. The effects of initial dye concentration, contact time, pH, equilibrium isotherm modelling and mechanism of dye adsorption in a binary system onto QKCF were studied in a batch system, while operation parameters which include inlet dye concentration, flow rate, and bed height were studied in a fixed bed column system.

The results showed that the maximum percentage removal from the single system were 98.10%, 99.58%,99.63% and 99.60% for RR, RB, AB and AG dyes respectively. In addition, the equilibrium data were best represented by the Langmuir isotherm model with maximum adsorption capacity of 185.20, 294.12, 303.03 and 344.83 mg/g for RR, RB, AB and AG dyes respectively, and the kinetic data were found to follow the pseudo-second-order kinetic model. Moreover, the maximum percentage removal reached up to 97.8% and 99.60% for RR and RB dyes respectively from the binary solution, while the maximum percentage removal reached up to 99.96% and 99.60% for AB and AG dyes respectively from the binary solution.

The fixed bed column showed better performance with lower influent dye concentration, less flow rate of the influent and higher adsorbent bed depth.

Overall, the present study showed that QKCF is a potential adsorbent for anionic dye removal from aqueous solutions either in a batch or a fixed bed column system.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PENGNYAHAN PEWARNA REAKTIF DAN ASID DARIPADA LARUTAN TUNGGAL DAN BINARI SECARA PENJERAPAN TERHADAP SERABUT TERAS KENAF TERKUATER

Oleh

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Ogos 2017

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Dianggarkan bahawa setiap tahun 280,000 tan pewarna teksti telah dilepaskan ke dalam efluen kilang tekstil, dan malangnya semua kilang masih menggunakan saliran air untuk membuang air efluen kilang. Dalam loji rawatan air sisa, karbon teraktif adalah penjerap yang digunakan secara meluas untuk menghapuskan pelbagai jenis pewarna, tetapi ia sangat mahal. Oleh itu, penjerapan dengan menggunakan berbagai jenis sisa-agro merupakan salah satu bahan alternatif untuk mengnyahkan pelbagai jenis pewarna dari larutan.

Dalam kajian ini, baki serabut teras kenaf (KCF) telah diubahsuai secara kimia dengan (3-kloro-3-hidroxypropil) trimetilammonium klorida (CHPTAC) untuk mengubah sifat-sifat permukaan bagi memerangkap pewarna anion dari larutan akueus. Kajian penjerapan sekelompok telah dijalankan untuk mengkaji prestasi serabut teras kenaf terkuater (QKCF) untuk mengnyahkan RB-reaktif merah, Reaktif Hitam-5, Asid Biru-5, dan Asid Hijau 25 dari sistem tunggal. Pelbagai parameter seperti kepekatan awal pewarna, dos penjerap, kelajuan pengadukan, masa sentuhan, suhu dan pH telah disiasat untuk sistem larutan tunggal tersebut.

Penyelidikan ini menerokai kesesuaian serabut teras kenaf terkuater (QKCF) untuk bertindak sebagai penjerap bagi mengnyahkan pewarna anion daripada sistem binari. Kesan kepekatan awal pewarna, masa sentuhan, pH, model keseimbangan isoterma dan mekanisma penjerapan pewarna dalam sistem binari ke atas QKCF telah dikaji dalam suatu sistem berkelompok, manakala parameter operasi termasuk kepekatan pewarna masuk, kadar aliran, dan ketinggian dasar telah dikaji dalam suatu sistem turus dasar tetap.

Hasil kajian menunjukkan bahawa peratusan maksimum pengnyahan dari sistem yang tunggal adalah 98.10%, 99.58%, 99.63% dan 99.60% bagi pewarna RR, RB, AB dan AG masing-masing. Tambahan pula, data keseimbangan adalah terbaik diwakili oleh model isoterma Langmuit dengan kapasiti penjerapan maksimum sebanyak 185.20, 294.12, 303.03 dan 344.83 mg/g untuk pewarna RR, RB, AB dan AG masing-masing, dan data kinetik didapati mengikuti model kinetik arahan-kedua-pseudo. Di samping itu, peratusan maksimum pengnyahan mencecah sehingga 97.8% dan 99.60% bagi pewarna RR dan RB masing-masing dari larutan binari, manakala peratusan maksimum pengnyahan mencecah sehingga 99.96% dan 99.60% bagi pewarna AB dan AG masing-masing dari larutan binari.

Turus dasar tetap menunjukkan prestasi yang lebih baik dengan kepekatan pewarna masuk yang lebih rendah, kurang kadar aliran masuk dan kedalaman dasar penjerap yang lebih tinggi.

Secara keseluruhan, kajian ini menunjukkan bahawa QKCF merupakan penjerap yang berpotensi untuk mengnyahkan pewarna anion daripada larutan akueus sama ada dalam kelompok atau pun sistem turus dasar tetap.

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Intidhar Jabir Idan Al-Tharwan
August 2017

I certify that a Thesis Examination Committee has met on 16 August 2017 to conduct the final examination of Intidhar Jabir Idan Al-Tharwani on her thesis entitled "Removing Reactive and Acid Dyes from Single and Binary Solutions by Adsorption on Quaternized Kenaf Core Fibers" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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Declaration by graduate student

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

Symbols	Description	Unit
C_o	Initial dye concentrations	mg/L
C_e	Equilibrium dye concentrations	mg/L
W	Weight of adsorbent	g
V	Volume of dye solution	L
q_e	Adsorption capacity at equilibrium	mg/g
q_t	Adsorption capacity at any time t	mg/g
q_{max}	Maximum adsorption capacity	mg/g
$q_{e,cal}$	Calculated adsorption capacity	mg/g
$q_{e,exp}$	Experimental adsorption capacity	mg/g
K_b	Adsorption equilibrium Langmuir constant	L/mg
K_L	Langmuir constant	L/g
K_{id}	Intraparticle diffusion rate constant	mg/g min
K_F	Freundlich constant	$(mg/g)(L/mg)^{1/n}$
R_L	Separation factor	Dimensionless
1/n	Surface heterogeneity	Dimensionless
R	universal gas constant	8.314 J/mol K
T	Absolute temperature	^oK
B	Temkin constant related to heat of sorption	j/mol
A	Temkin isotherm equilibrium binding constant	L/g
n	number of data points	-
ΔG^o	Change in standard free energy	kJ/mol
ΔH^o	Change in enthalpy	kJ/mol

ΔS^o	Change in standard entropy	j/mol K
K_1	Pseudo-first-order adsorption rate constant	1/min
K_2	Pseudo-second -order rate constant	mg/g min
D_{eff}	effective diffusivities value for the homogeneous model	m^2/s
Ds	Surface diffusivity	m^2/s
D_{Pa}	Pore diffusivity	m^2/s
ϵ	Porosity of adsorbent	-
C_1	Required dye concentration	mg/L
C_2	Stock dye concentration	mg/L
V_1	Required dye volume	mL
V_2	Stock dye volume	mL
Q	Flow rate	mL/min
m	Amount of adsorbent in the column	g
K_{TH}	Thomas rate constant	mL/(mg.min)
K_{YN}	Yoon Nelson constant	min^{-1}
K_{AB}	Adams Bohart constant	L/(mg.min)
q_o	Thomas constant for bed capacity	mg/g
τ	Time required for 50% adsorbate breakthrough	Min
U_o	Linear velocity	cm/min
N_o	Maximum dye uptake capacity per unit volume of adsorbent column	mg/L

LIST OF ABBREVIATIONS

KCF	Kenaf core fiber
MKCF	Mercerized Kenaf core fiber
QKCF	Quaternized kenaf core fiber
CHPTAC	(3-chloro-2-hydroxypropyl) trimethylammonium chloride
RR	Reactive Red-RB dye
RB	Reactive Black 5 dye
AB	Acid Blue 25 dye
AG	Acid Green 25 dye
INTROP	Institute of Tropical Forestry and Forest Product
SEM	Scanning Electron Microscope
EDX	Energy Dispersive X-Ray
BET	Brunauer-Emmett-Teller method
FTIR	Fourier Transform Infrared Spectroscopy
TGA	Thermogravimetric analysis
SSE	Sum of the Squares Errors
MTZ	Mass Transfer Zone

CHAPTER 1

INTRODUCTION

1.1 Introduction

The colour is the essential attractive thing for any marketing products. regardless of its arrangement, if the colour of the product is inappropriate, then the sales might not be good either. Dyes are used firstly in the production of customer products, inclusive textiles, sheet, plastics, and pigments. They add dyes and styles to materials (Kant, 2012). A textile was previously being coloured with naturalistic dyes. However, these provided a restricted and a tedious scope of colours. In addition, they showed less colour firmness once subjected to sunlight or washing. For that reason, a mordant type of dye compound was needed in order to fasten the fiber as well as the dye with each other, thus creating the dyers' job tiresome (Whitaker and Willock, 1949). The introduction of artificial dyes from petroleum origin in the late 19th century finished the market for natural dyes from a plant source, which had been in use since 3500 BC. Nowadays, more than 100,000 various dye structures have been synthesized (Kant, 2012). Synthetic dyes have supplied colours which dye quick and produce a broad colour range, but such dyes are said to have toxic nature that make environmentalists seriously worried. Using artificial dyes have, in fact, a reverse impact on various types of existence (Priya, 2015).

Over 3600 types of individual textile dyes are being made by the manufacturers in the present. Throughout textile manufacture's processes, it is estimated that 8000 different type of chemicals is used for both printing and dyeing operations. Around 1.6 million litters of water per day is spent in a textile mill which has a fabric production of around 8000 kg/day, 8% of this water is consumed in printing and 16% in dyeing (Ravi et al., 2014). For this kind of dye, water used for dyeing differs from 30 - 50 litters per kg of cloth. As a result, the contribution of dyeing section is about 15% - 20% of the total flow of effluent. According to The World Bank, 17% -20% of industrial water pollution occurs as a result of textile dyeing and treatment. This vast water use is a key of water body pollution (Rajan et al., 2015).

Dyes can be distributing into three mainly classes: (a) cationic (all basic dyes), (b) anionic (direct, acid, and reactive dyes), and (c) non-ionic (dispersed dyes) (Greluk and Hubicki, 2010). Cationic dyes are artificial dyes acting as bases and once soluble in water, forming a colored ion salt, which will then react with the anionic sites on the surface of the substrate. Anionic dyes have negative ions due to the surplus presence of the OH^- ions in solution. Reactive and basic dyes have favourite characteristics of bright colour, easily dissolving in water and applying to fabric, moreover they are inexpensive. All these made them widely used in the textile mills (Karadag et. al., 2007).

The researchers in effluent textile treatment have often centred on reactive dyes for these reasons: (i) they are used to dye cotton fibers, and since about half of the world's fiber consumption is of this fiber type, textile factories use more than 80000 tonnes of reactive dyes every year. (Chakraborty et al., 2005). (ii) the dye hydrolysis in alkaline dye bathtub results in wasting about 30% of the reactive dyes used, and (iii) the traditional wastewater treatment plants are not good enough to remove anionic dyes, including reactive dyes, which cause, in turn, coloured water-paths (Kyzas et. al., 2012). Table 1.1 shows the characteristic features of dyes used in the textile dyeing process (Adamu, 2008).

Table 1.1 : Characteristics of dyes used in textile dyeing operations (Adamu, 2008)

Type of dye	Fixation (%)	characterization	species of pollutants related with the dyes
Reactive	less than 80	water-soluble, anionic composite, greatest dye group	Colour, salt, unfixed dyes, alkali
Acid	less than 93	water-soluble, anionic composite	Colour, unfixed dyes, and organic dyes
Direct	less than 95	water-soluble, anionic composite, can be applied (without mordant or metals like chromium and copper) directly to celluloses	Salt, colour ,unfixed dyes, copper salts, cationic fixing agents
Basic	less than 98	water-soluble, used with a mordant	Unfixed dyes, alkali
Vat	less than 95	ancient dyes, more chemically complex, water-insoluble	Alkali, reducing agent , oxidizing agent
Disperse	less than 92	water-insoluble	Colour, carriers, reducing agent, organic acids
Sulfur	less than 70	organic compounds containing sulfur or sodium sulfide	Alkali, colour ,oxidizing agent, reducing agent, unfixed dyes

The most challenging mission in wastewater treatment plants are the removal of anionic dyes, because they are water-soluble which have acidic properties and produce very shining colours in water. It has been estimated that the total dye consumption in textile industry around the world is more than 10,000 tonnes annually and about 10-15% of these dyes are discharged as wastewater through the dyeing processes (Gupta et al., 2013).

A colour of the textile effluent (ADMI colour value - American Dye Manufacturer Institute colour value), and hazardous organic compounds or other potentially dangerous included into each textile processing stages are recorded in Table 1.2 (Cooper, 1995).

Table 1.2 : The main characteristics of a cotton wet processing wastewater (Cooper, 1995)

Process	Colour (ADMI)	BOD, g O_2/L	COD, g O_2/L	pH	TDS, g/L	Water consume/ Kg of product (L)
Dyeing	1450-4750	0.01-1.8	1.1-4.6	5-10	0.05	8-300
Desizing	-	1.7-5.2	4.6-5.9	-	-	3-9
Mercerization	-	0.05-0.1	1.6	5.5-9.5	4.3-4.6	232-308
Scouring	694	0.1-2.9	8.0	10-13	-	26-43

Water stream contaminated by industry effluent is regarded as one of the most common problems in the world. The presence of colour in water has ever been undesirable for any purposes. The presence of metals in different dyes is fundamental for their function as textile colourants. Unavoidable, these toxic metals will reach to aquatic environments and pose a significant menace to the lives in the aquatic system (Waranusantigul et al., 2003). For example, the heavy metals associated with acid dyes are lead, cobalt, zinc, copper, and chromium. While, the heavy metals associated with Reactive dyes are lead, copper, and chromium. Discharge of these dyes with associated toxicity metals may cause negative hazards impacts to the ecosystem and human health (Hameed et al., 2008; Verma, 2008). Dyes that reach to water bodies absorb and reflect the sunlight inhibit photosynthesis, and thus, disturbs aquatic life and, consequently affects food chain (Bouasla et al., 2010). Such dyes are considered carcinogenic and toxic, and therefore, form serious threat to watery life. Synthetic dyes are composed of complex aromatic molecular structures that are constructed to resist for many agents like exposure to venation, laundry, water, light, and all these make them high stabilization. As a result, dyes are resistance to remove by conventional water treatment plants (Indrani et al., 2016). Thus, increasing levels of dyes pollutants in the environment not only cause a dangerous menace to the environment but also affect the public health. To deal with this problem, industrial effluents must be treated prior to discharging.

Adsorption process can remove or reduce different kinds of pollutants including dyes and is widely used in wastewater treatment plant (Hamdaoui, 2006). Various adsorbents have been attempted to remove various types of synthetic dyes. In wastewater treatment plant, coal-based activated carbon is a widely used adsorbent to remove many types of dyes, but it is very expensive because original material is costly and required high energy for the activation method. Moreover, there are many problems associated with the regeneration of activated carbon (Gupta et al., 2013). Therefore, numerous studies have been focused on low-cost and efficient adsorbents materials derived from natural materials for the removal various species of dyes from an effluent (Gupta and Suhas, 2009). Following this tendency, cellulose-based layer plants are seen as encouraging materials for the elimination of multi-kinds of pollutants from wastewater. Agricultural biomass plants are one of the plentiful substances, cheap and renewable natural material (Vismara et al., 2009). In this context, the present research deals with the activity of removing acid and reactive anionic dyes from aqueous solution by using inexpensive and native agricultural biomass.

1.2 Problem Statement

It is estimated that every year 280,000 tonnes of textile dyes are released in textile mill effluent (Jin et al., 2007), and unfortunately, all factories are still using water streams for discharging their effluent water. However, the necessity to the renewal of our water resources has received growing interest. This has led up to the evolution of strategies to, reversion water to its source in the least possible pollution form, to enable use water again. These strategies and processes are termed as "wastewater treatment".

Effluent from textile industries should meet the Environmental Quality (Industrial Effluent) Regulations 2009. Any wastewater treatment plant has to design and able to treat the wastewater to meet the standard specifications (Kharat, 2015).

A numerous number of research papers have considered adsorption processes for single component systems. However, due to the complexity of the textile effluent and the variability of the dyeing process little successes have been reported in using this technique as a full-scale process to decolourise textile wastewater. Hence, this study was undertaken to address the problems associated with multi-component adsorption from aqueous solutions. This is essential for the accurate design of adsorption systems as the effect of multicomponent interactions in the process effluent may cause deterioration in the adsorption capacity of an adsorbent for dyes.

The use of commercial activated carbon for removing dyes is expensive as it is obtained from non-renewable starting materials like lignite, coal and petroleum coke. Therefore, aqueous phase adsorption by utilizing different types of agro-residues is one of the most alternatives materials (Kharat, 2015). Agricultural biomass can be either procured directly from plant species or indirectly from a processing of domestic, commercial, industrial or agricultural products (Viglasky et al., 2009). Last few decades, interest on producing adsorbent which can be derived from renewable, abundant, and low-cost substances generated from an agricultural origin was increased (Babel and Kurniawan, 2003). As compared to the production of activated carbon which requires high energy consumption (steam or gasification processes), a chemical modification process on agricultural biomass, such as quaternization, which requires less energy (no gasification) to produce adsorbent, is considered. There are some reported literatures about quaternized adsorbents, however, no research has ever reported on the quaternized kenaf core fiber (KCF) as adsorbent for binary dyes removal from aqueous solution to date. Therefore, the study on development of quaternized KCF as a novel adsorbent in removing binary dyes in aqueous solutions is needed.

1.3 Research Objectives

The following objectives were addressed in this work:

1- To modify and optimize the chemical modification process of kenaf core fiber (KCF) by using quaternized agent; (3-chloro-2-hydroxypropyl) trimethylammonium chloride.
2- To examine the removal of anionic Reactive Red RB, Reactive Black 5, Acid Blue 25 and Acid Green 25 dyes from aqueous solutions via single dye batch adsorption systems using the quartenized kenaf core fiber (QKCF).
3- To investigate the removal of binary reactive dyes (Reactive Red RB and Reactive Black 5) and binary acid dyes (Acid Blue 25 and Acid Green 25) from aqueous solution via batch and fixed-bed column adsorption systems using the quartenized kenaf core fiber (QKCF).
4- To analyze the equilibrium isotherm models using Langmuir, Freundlich and Temkin models for single dye system and also an examination of the applicability of the extended Langmuir model and Jain-Snoeying model for multicomponent dyes in binary systems.

1.4 Scope of Research Study

In this study, kenaf core fiber (KCF) was chemically modified by the quaternized agent and used as adsorbent to adsorb RR, RB, AB and AG dyes from single and binary aqueous solution in batch and continues studies and the results from this study were compared with other research which used modified agricultural waste. The influencing operating factors like agitation speed, temperature, pH, initial dye concentration, adsorbent dosage and contact time were examined in a batch mode system. While, operation parameters which include inlet dye concentration, flow rate, and bed height were studied in fixed bed system. The adsorption isotherm, kinetics and thermodynamic studies of dye adsorption were also studied in detail.

1.5 Novelty of Research Study

In practice, most industrial wastewaters contain more than one dye (as a mixture). However, to date, the utilization of quartenized kenaf core fiber (QKCF) in batch and fixed-bed adsorption studies to adsorb binary reactive dyes and binary acid dyes has not been reported elsewhere. Thus, in the present work, investigation on adsorption in binary system was carried out. Pre-treatment with different concentration of Sodium hydroxide solution was used to increase the accessibility and chemical reactivity of QKCF to obtain the cationic cellulose with ideal adsorption ability for removing anionic dyes. The mechanism of dyes adsorption onto QKCF was also studied in detail.

1.6 Thesis Layout

There are a total of six chapters in this thesis. Chapter One (Introduction) gives the overview of present situation of water pollution problem. This chapter briefly explains the research objectives and overall content of the thesis.

Chapter Two (Literature Review) provides important information about preparation of adsorbent. Theoretical backgrounds of batch adsorption study for adsorbate-adsorbent system are explained in the second section. The third section summarizes the sorption dynamics of fixed bed in terms of Thomas, Adams-Bohart, and Yoon-Nelson models.

Chapter Three (Materials and Methodology) deals with the experimental set up for production of adsorbent. The experimental procedure for single system in batch adsorption studies, binary system in batch adsorption studies, fixed-bed adsorption studies for binary system and regeneration of the prepared adsorbent are explained. The last section of the chapter comprises of schematic flow chart of all research activities in this project.

Chapter Four (Results and Discussion) presents the results obtained for surface characterization of the prepared adsorbents by different physical and chemical tests. The results obtained from batch adsorption studies for adsorbent dyes on QKCF from single solution were presents in second section. Equilibrium isotherm, kinetic studies and thermodynamic parameters are evaluated in in third section. Overall performance of QKCF towards the anionic dyes under investigation is compared with other types of adsorbents.

Chapter Five (Results and Discussion) deals with the batch adsorption studies carried out for dyes in binary system. Equilibrium isotherm and kinetic parameters are evaluated in second section. The break through curve analysis and the model parameters necessary to understand sorption dynamics in continuous flow adsorption were evaluates in third section. The last section provides the necessary information for regeneration of the dyes loaded adsorbent by using eluting agent.

The conclusions of the overall findings of this research are given in Chapter Six. The conclusion summarizes the listed of the objectives are achieved throughout this study. The last section deals with some recommendations and their significance related to this study for future application.

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