UNIVERSITI PUTRA MALAYSIA

BATCH AND FIXED BED ADSORPTION STUDY FOR REMOVAL OF ACID AND REACTIVE DYES FROM AQUEOUS SOLUTION USING MODIFIED KENAF CORE

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By

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

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DEDICATION

This thesis is dedicated to my brother Habib, my son Mohammed, my wife, and my family and supports. Without them, none of this would have been possible.

Maytham Kadhim Obaid

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Master of Science

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Chairman : Professor Luqman Chuah Abdullah, PhD
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The extensive use of commercial activated carbon as an adsorbent for the purification of industrial effluent from textile industry is not economically feasible due its high operational cost. Therefore, this research has been undertaken to explore the potential of certain agro based residues to produce suitable adsorbent.

In this research, evaluation of the removal of reactive orange16 (RO16) and acid red 114 (AR114) dyes from aqueous solution was studied in batch and fixed bed column system by using modified kenaf core fibre (MKCF), in which kenaf being one of the agricultural crops used as low-cost adsorbents instead of activated carbon. In this study, kenaf core fibre (KCF) was modified successfully by treating with (3-chloro-2-hydroxypropyl)- trimethylammonium chloride (CHMAC) as quaternization agent.

In batch system, Results showed that the maximum removal of RO16 and AR114 were 97% and 99% respectively. Moreover, the results obtained from the isotherm studies were found to be best fitted with Freundlich isotherm with correlation coefficients (R^2) of 0.9924 for RO16, and isotherm studies were best fitted with Langmuir isotherm with correlation coefficients (R^2) 0.9853 for AR114. It was found that the kinetic study followed the pseudo-second-order kinetic model with correlation coefficients R^2=0.9997 and 0.9953 for RO16 and AR114 respectively and the maximum adsorption capacity for RO16 and AR114 were 416.86 mg/g and 238.56 mg/g, respectively.

The fixed bed column experiment was carried out by varying the flow rates, initial concentration of the dyes and the height of the bed. experimental data analysis by using three models Thomas, Yoon- Nelson and Adam- Bohart, It was found that the best color removal percentage increased when there was a decrease in the dye

concentration, low flow rates and an increase in the bed depth for the both dyes studied. The experimental results have shown a good agreement with the theoretical results. This study showed that the low-cost kenaf fibres could be used for dye removal.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

KAJIAN PENJERAPAN KELOMPOK DAN LAPISAN TETAP UNTUK PENYINGKIRAN PEWARNA ASID DAN REAKTIF DARI LARUTAN AKUEUS DENGAN MENGGUNAKAN SERAT TERAS KENAFYANG DIUBAHSUAI

Oleh

MAYTHAM KADHIM OBAID AL- MAMOURI

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Penggunaan secara meluas karbon komersil yang diaktifkan sebagai bahan penjerap bagi penulenan bahan efluen industri adalah tidak begitu sesuai secara ekonomi kerana kos operasinya yang tinggi. Oleh itu, kajian ini telah dijalankan bagi meneroka potensi sisa pertanian tertentu untuk menghasilkan bahan penjerap yang bersesuaian

Dalam cubaan ini, penilaian penyingkiran pewarna orange16 reaktif (RO16) dan merah asid 114 (AR114) daripada larutan akueus dikaji dalam model kumpulan dan turus dasar tetap dengan menggunakan serat teras kenaf diubahsuai (MKCF), di mana kenaf merupakan salah satu daripada tanaman pertanian yang digunakan sebagai bahan penjerap kos-rendah menggantikan karbon teraktif. Di dalam usaha ini, serat teras kenaf (KCF) telah diubahsuai dengan jayanya secara merawat dengan (CHMAC) sebagai ejen kuartener.

Hasil kajian menunjukkan bahawa penyingkiran maksimum RO16 dan AR114 ialah 97% dan 99% masing-masing pada 30ºC. Selain itu, keputusan yang diperolehi daripada kajian isoterma telah dipadankan terbaik dengan isoterma Freundlich dengan pekali korelasi sebanyak (R^2) 0.9924 untuk RO16, dan kajian isoterma telah dipadankan terbaik dengan isoterma Langmuir dengan pekali korelasi (R^2) 0.9853 untuk AR114. Didapati bahawa kajian kinetik mengikuti model kinetik pseudo-order-kedua dengan pekali korelasi R2= 0.9997 dan 0.9953 untuk RO16 dan AR114 masing-masing; kapasiti penjerapan maksimum bagi RO16 dan AR114 adalah 416.86 dan 232.56 masing-masing.

Ujian penjerapan untuk menentukan kadar penyerapan pewarna reaktif dan berasid. di dalam system turus juga telah dijalankan Eksperimen turus telah dijalankan dengan mengubah kadar aliran, kepekatan awal pewarna dan ketinggian dasar pada. Didapati bahawa peratusan penyingkiran warna meningkat apabila terdapat penurunan kepekatan pewarna, kadar aliran yang rendah dan peningkatan kedalaman dasar untuk kedua-dua pewarna yang dikaji. Dalam eksperimen, kadar aliran turus dikekalkan pada. Kajian ini telah menunjukkan bahawa serat kenaf kos-rendah boleh digunakan untuk penyingkiran pewarna. Kajian penjanaan semula telah dijalankan dan bahan penjerap itu didapati boleh digunakan semula dengan penurunan minimum dalam kapasiti penjerapannya.

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January 2017

This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) were adhered to.

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LIST OF ABBRIVATIONS

NKCF	Natural kenaf core fibre
MKCF	Modify kenaf core fibre
CHMAC	(3-chloro-2-hydroxypropyl)trimethylammonium chloride
RO16	Reactive orange16
AR114	Acid red114
SSE	Sum of the Squares of the Errors

LIST OF SYMBOLS

q_e	Amount of dye adsorbed per gram of adsorbent at equilibrium
q_m	Maximum adsorption capacity per gram of adsorbent
q_t	Amount of dye adsorbed per gram of adsorbent in time
R^2	Correlation coefficient
C_e	Concentration of solute in solution at equilibrium
C_o	Initial concentration of solute in solution
q_{max}	Equilibrium adsorption capacity (mg/g)
R_L	Dimensionless constant separation factor
K_L	Langmuir constant
K_F	Freundlich constant
1/n	an empirical parameter that depends on adsorption rigour
R	constant of gas 8.314 J/mol
T	Absolute temperature
B	Heat of adsorption J/mol
A	Temkin isotherm constant L/g
Qt	Value of adsorbed at a given time (min)
K_1	Average constant of adsorption operation (1/min) (pseudo-first-order)
k_{id}	is the intra-particle diffusion rate constant (mg/g min1/2)
C	is the intercept
V_{eff}	effluent volume
K_{TH}	Thomas model constant (L/min.mg)
K_{YN}	Yoon-Nelson rate constant (1/min)
K_{AB}	Kinetic constant of Adams–Bohart model (L/mg min)

N_o	saturation concentration from Adams–Bohart model (mg /L)
Z	bed depth of column (cm)
F	linear velocity (cm/ min)

CHAPTER 1

INTRODUCTION

1.1 Water pollution

The problems of environmental pollution, specifically water pollution that affects the aquatic environment, have been an area of major concern for decades now. Water is considered to be the foremost reason for the existence of life on earth. The history of human civilisation shows that all communities live near to water sources. Humans can survive and obtain healthy lives; healthy life is the presence of fresh and clean water (Gupta et al. 2016). In recent years, pollution has become a glaring issue across the world. Pollution is highly detrimental to the environment irrespective of whether it is caused by human activities or natural processes. Water pollution can be regarded as the most crucial environmental pollution problem. It damages the aquatic ecosystems and causes health hazards to those who depend on that polluted water. Industries such as tanning, textile, printing, plastic, and carpet, are some of the main sources of water pollutants (Zurhana et al., 2015). These industries require huge amounts of water for their various operations (Kayode et al., 2015). The contaminated water coming out of these industries is then discharged into rivers, ponds and other water bodies, without any serious care or processing. This is more the case in developing countries where there is a serious lack of strict laws related to environment protection, or the laws that are there have no enforcement at all, which in turn pollute the clean water supplies (UN Water, 2009). In some regions, 50% of the different species of fish that live in freshwater are on the verge of extinction and almost one-third of the amphibians are likely to be extinct (Vié et al., 2009). The rate of extinction of freshwater fishes is five times greater than that of other types (Ricciardi et al., 1999). Even in the developed world, in countries like France, the quality of drinking water is not assured. On testing the quality of drinking water of 3 million people in France, it was found that the quality of drinking water does not match up to the required quality recommended by WHO. It was uncovered that about 97% of the underground water samples do not satisfy the required level for nitrate (UN Water, 2009). In recent years, the concentration of dyes in the contaminated water might reach a level as high as 15% (Shamel, et al. 2016).

1.2 Kenaf

Kenaf is a member of the hibiscus family which it is related to cotton, hemp, and okra. Kenaf has a high growth rate, rising to heights of 12 to 18 feet’s in about 4 to 5 months. Its yield of 6 to 10 tons and for new varieties may reach 12 tons of dry weight per acre per year. The Kenaf production in Malaysia is estimated between 3,000 tons to 10,000 tons kenaf fibre annually (Lansah at al., 2016). Which is most likely a native of Southern Asia, Kenaf is also cultivated in Bangladesh, Malaysia, Indonesia, South Africa, Thailand, Vietnam, parts of Africa, and to a small extent, in south-east Europe for its fibre(Wilson et al. 1965).

The key uses of kenaf fibre include the manufacturing of ropes, twines, and coarse clothes. Most of the kenaf fibres are used for animal bedding and feed (Webber, 1993). That could be used as thermal insulators or acoustic insulators for buildings or vehicles. These composites find their use in paper and garment industries due to their property of slow ignition (Rajappan et al., 2015).

1.3 Adsorption of pollutants by using kenaf fibre

Much attention has recently been focused on various biosorbent materials used as an adsorbent for the removal of dyes from aqueous solutions. Natural fibre has become apparently important as a source of absorbent. Kenaf one of the best adsorbent was used to remove a lot of dyes and heavy metals. Table 1.1 illustrate kenaf core fibre as adsorbent.

Table 1.1 : Utilisation of kenaf core fibre as adsorbent

Material	References
Nickel	Annie et al., 2015
Fluoride	Yusof, 2015
Waxes	Wong, et al., 2013
Lead, Mercury, Copper, Zinc and Cadmium	Dinesh, 2009
Pb (II) and Cr (III) (cationic heavy metals)	Marcus, 2013
Copper (II)	Hasfalinaa et al., 2012

1.4 Problem statement

The extensive use of commercial activated carbon as an adsorbent for the purification of industrial effluent from textile industry is not economically feasible due its high operational cost. Hence, this research has been undertaken to explore the potential of certain agro based residues to produce suitable adsorbent, where the thermal activation process is omitted. (Koay et al., 2014).

Environmental pollution is one of the most significant problems of modern times. The contaminant water from the textile contains hazardous synthetic dyes (Lin et al., 2016). The release of synthetic dyes into water bodies after various industrial operations is unavoidable. This causes severe pollution problems for the environment.

The removal of synthetic colors from the wastewater is usually considered more necessary because it is the synthetic colors that damage the water environment and pose risks to human health (Sadaf et al. 2014). There are several methods of treatment; including chemical, physical and biological, chemical modification on biomass such as quaternisation to produce adsorbent is the solution to this problem.

Adsorption is considered to be the best method for removing dyes from solutions due to its simplicity of design, cost-effectiveness, high efficiency, high availability and its potential to separate a wide range of chemical compounds (Mashhadi et al., 2016). Kenaf is a widely cultivated crop, particularly in the tropics. Kenaf fibers have high mechanical strength high percentage of cellulose which makes it favorite for modified processing to producing adsorbent. Chemical modification of KCF is better than other adsorbents because it is prepared with a minimum of processing and without thermal treatment. Widely used to separate chemical compounds. High efficiency of adsorption

1.5 Objectives

This research is to utilise a locally obtainable material as adsorbent rather than commercial activated carbon for enhancing the quality of industrial effluent and offering further treatment before releasing into drains or other water or drains or for industrial and agricultural uses. The recommended adsorbent (agriculture harvests) is likely to be quite inexpensive in comparison to other materials like carbon. It is available in Malaysia and possibly less costly to prepare, and thus operating costs can be significantly controlled.

1. To prepare and characterise modified kenaf core fibres (MKCF) adsorbent by using a quaternisation agent (3-chloro-2-hydroxyproply) trimethylammonium chloride (CHMAC)
2. To analyses the viability of using natural and modified kenaf core fibres by quaternisation as adsorbents to eliminate reactive orange 16 and acid red 114 dyes from aqueous solutions.
3. To study the adsorption isotherm and kinetics, by using batch analysis and fixed-bed column analysis.

1.6 Scope of work

In this study, an attempt was made to quaternizing kenaf core fibre and uses it as an adsorbent to adsorb RO16 and AR114 dyes from aqueous solution. The effect of MKCF, pH, dosage, contact time, temperature, speed and concentration studied in detail. In batch, the adsorption isotherm study by using Freundlich, Langmuir and Temkin kinetics studies by used Pseudo-first order, Pseudo-second-order dye and Intraparticle effective diffusivity were studied. In fixed bed column adsorption experiment Parameters: Effect of flow rate, height and concentration. The experimental data Analysis by using Thomas, Yoon-Nelson, and Adams-Bohart models.

1.7 Organisation of thesis

This thesis is made up of 5 chapters in Chapter 1, (Introduction) covers water pollution, textile dyes effluent, history of Kenaf, uses, Kenaf in Malaysia, problem statement, adsorption of pollutants by using kenaf fibre, objectives of the study, scope of this work, and organisation of the thesis. Chapter 2, (Literature Review) encompasses the acid dye, reactive dye, textile dye effluent treatment. The batch study covers the Langmuir model, Freundlich model and Timkin model, as well as kinetic and intraparticle effective diffusivity. Column study encompasses the Yoon–Nelson method, Adams-Bohart method, and Thomas method. Chapter 3, (Materials and Methods), we have presented the comprehensive list of materials and methods used in our study. Chapter 4, (Results and Discussion), the results and the collected data have been analysed. Chapter 5, (Conclusion) concludes all the observations as well as offers recommendations for future studies.

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