ACTIVATED CARBON FROM LEMON PEEL AND MANGOSTEEN PEEL FOR DYES REMOVAL

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ACTIVATED CARBON FROM LEMON PEEL AND MANGOSTEEN PEEL FOR DYES REMOVAL

by

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LIST OF ABBREVIATIONS

AC	Activated Carbon
AP	Adequate Precision
ANOVA	Analysis of Variance
BET	Brunauer-Emmett-Teller
CAC	Commercial activated carbon
CCD	Central Composite Design
FTIR	Fourier Transform Infrared
IR	Impregnation ration
IUPAC	International Union of Pure and Applied Chemistry
LP	Lemon peel
LPAC	Lemon peel based activated carbon
MB	Methylene blue
MP	Mangosteen peel
MPAC	Mangosteen peel based activated carbon
RBBR	Remazol brilliant blue R
rpm	Rotation per minute
RSM	Response surface methodology
SEM	Scanning electron microscopy
SST	Sum of squares
STA	Simultaneous thermogravimetric analyzer

LIST OF SYMBOLS

		Unit
$\pm\alpha$	Distance from central point	-
A	Arrhenius factor	-
A_i	Measured absorbance for component i	-
A_T	Temkin equilibrium binding constant	L/mg
b_c	Path length of the cell	cm
b_i	Linear coefficient	-
b_{ij}	Quadratic coefficient	-
b_o	Constant coefficient	-
B_T	Temkin constant related to free energy	L/mg
C_e	Concentration of adsorbate at equilibrium	mg/L
C_o	Initial adsorbate concentration	mg/L
C_t	Concentration of adsorbate at time t	mg/L
D_p	Average pore diameter	nm
E_a	Arrhenius activation energy of adsorption	kJ/mol
k	Number of variables	-
k_1	Pseudo-first order rate constant	1/min
k_2	Pseudo-second order rate constant	g/mg.h
K_F	Freundlich constant related to free energy	$mg/g(L/mg)^{1/n}$
K_L	Langmuir constant related to free energy	L/mg
M	Mass of adsorbent	g
M_1	Concentration of stock solution	mg/L
M_2	Concentration of diluted adsorbate solution	mg/L
N	Total number of experiments/data point	-
n_F	Freundlich constant	-
Q_m	Langmuir monolayer adsorption capacity	mg/g

q_e	Amount of adsorbate adsorbed at equilibrium	mg/g
q_t	Amount of adsorbate adsorbed at time t	mg/g
$q_{t, cal}$	Calculated adsorption uptake at time t	mg/g
$q_{t, exp}$	Experimental adsorption uptake at time t	mg/g
R	Universal gas constant	J/mol.K
R^2	Correlation coefficient	-
R_L	Langmuir dimensionless separation constant	-
S_{BET}	BET surface area	m^2/g
T	Absolute temperature	K
V	Volume of solution	L
V_1	Volume of stock solution	mL
V_2	Volume of diluted adsorbate solution	mL
V_{meso}	Mesopore volume	cm^3/g
V_T	Total pore volume	cm^3/g
W_c	Dry weight of AC	g
W_{char}	Dry weight of char	g
W_{KOH}	Dry weight of KOH pellet	g
W_o	Dry weight of precursor	g
x_1	Radiation power	Watt
x_2	Radiation time	min
x_3	Impregnation ratio (IR)	-
x_i	Coded value of variables	-
x_j	Coded value of variables	-
y	Predicted response	-

Greek letters

$\Delta G°$	Changes in standard free energy	kJ/mol

$\Delta H°$	Changes in standard enthalpy	kJ/mol
$\Delta S°$	Changes in standard entropy	J/mol.K
Δqt	Normalized standard deviation	%
ε_λ	Molar absorptivity coefficient of solute at wavelength	-
λ	Wavelength	nm

KARBON TERAKTIF DARIPADA KULIT LEMON DAN KULIT MANGGIS UNTUK PENYINGKIRAN PEWARNA

ABSTRAK

Penjerapan pewarna sebagai teknik untuk merawat air kumbahan terutamanya dari industri tekstil dan pewarna kekal sebagai skop kajian yang menarik. Sebaliknya, penjerapan menggunakan penjerap boleh diperbaharui tidak memberikan masalah dan operasi secara komersial murah. Oleh itu, kajian ini bertujuan untuk menghasilkan karbon teraktif (AC) daripada kulit lemon (LP) dan kulit manggis (MP) untuk menyingkirkan MB dan RBBR daripada larutan akuas. AC menjalani proses pengaktifan fizikimia yang menggunakan rawatan kimia kalium hidroksida (KOH), penggasan karbon dioksida (CO_2) dan dipanaskan oleh gelombang mikro. Semasa penyediaan AC, syarat penyediaan optimum diperoleh daripada metodologi permukaan sambutan (RSM). Keadaan yang optimum adalah kuasa radiasi gelombang mikro, masa radiasi dan nisbah impregnasi (IR). Luas permukaan Bruneaur-Emmet-Teller (BET) dan jumlah isipadu liang yang tinggi diperoleh untuk sampel LPAC yang disediakan (730.25 m^2/g dan 0.3950 cm^3/g dan MPAC (892.10 m^2/g dan 0.4640 cm^3/g). Kesan kepekatan awal pewarna (25-300 mg/L), masa sentuhan (0-24 jam) dan suhu larutan (30-60 °C) turut dinilai. Data keseimbangan yang diperolehi bagi kedua-dua pewarna telah terbaik diwakili oleh model Freundlich dengan faktor Freundlich permukaan kepelbagaian, $1/n_F$ didapati 0,6579, 0,6944, 0,6452 dan 0,6459 untuk MB-LPAC, MB-MPAC, RBBR-LPAC dan RBBR-MPAC masing-masing Manakala, untuk analisa kinetik, didapati bahawa model pseudo-tertib-kedua dan pseudo-tertib-pertama adalah masing-masing paling sesuai digunakan untuk menentukan mekanisma penjerapan MB dan RBBR. Penjerapan MB dan RBBR yang diuji ke atas AC's adalah secara eksotermik

ACTIVATED CARBON FROM LEMON PEEL AND MANGOSTEEN PEEL FOR DYES REMOVAL

ABSTRACT

Dyes adsorption as a technique for treating wastewater especially from textile and dyeing industry remains an area of interest. On the other hand, adsorption using renewable adsorbent provide trouble free and commercially cheap operation. Therefore, this study aims to synthesise activated carbon (AC) from lemon peel (LP) and mangosteen peel (MP) to adsorb MB and RBBR dyes. AC's undergoes physiochemical activation process which involves potassium hydroxide (KOH) impregnation and carbon dioxide (CO_2) gasification and heated with microwave. During the preparation of AC's, the optimum preparation conditions were obtained from response surface methodology (RSM). The optimum parameters are microwave radiation power, radiation time and KOH: char impregnation ratio (IR). Relatively high Brunauer-Emmet-Teller (BET) surface area and total pore volume were found on prepared LPAC (730.25 m^2/g and 0.3950 cm^3/g and MPAC (892.10 m^2/g and 0.4640 cm^3/g). The significance of initial dye concentration (25-300 mg/L), contact time (0–24 hours) and solution temperature (30-60°C) were also studied. The obtained equilibrium data for both dyes were best fitted by Freundlich model with Freundlich surface heterogeneity factor, $1/n_F$ was found to be 0.6579, 0.6944, 0.6452, and 0.6459 for MB-LPAC, MB-MPAC, RBBR-LPAC and RBBR-MPAC respectively. Meanwhile, the kinetics data were best demonstrated by the pseudo second-order model for MB and RBBR. The adsorption process of MB and RBBR onto AC's were exothermic in nature.

CHAPTER ONE

INTRODUCTION

1.1 Textile industries

Wastewater from dyeing and textile industrial processes are known to consist of highly coloured species. More than 10 000 dyes with a total annual generation of 7 × 10^5 MT are readily obtainable. It is predicted that around 15% of the dye stuff are escaping in industrial effluents while production and processing stage (Pepe et al., 2013). Highly coloured wastes hinder light penetration and may disorder the ecosystem. These dyes reflect sunlight from entering the water, which then interferes and hinder photosynthesis in aquatic plant. Furthermore, dyes alone are poison to certain organisms. Methylene blue (MB) and remazol brilliant blue R (RBBR) have several usage in chemistry, biology, medical science and dyeing industries. Its long term exposure can cause nausea, anaemia, vomiting and hypertension (Foo and Hameed, 2012).

1.2 Industrial dye effluent treatment

Several chemicals, physical and biological methods, including adsorption, absorption, membrane filtration, ozonation, advanced oxidation, liquid-liquid extraction and coagulation/ flocculation have been broadly applied for the wastewater treatment. Although chemical treatment using coagulating agent is a robust way for dyes removal, it is not economical feasible for large scale industries operation as the accumulation of concentrated sludge can create further disposal problems such as transportation cost and environmental issue (Shamsuddin et al., 2016).

Adsorption is a capable separation method with regard to simplicity of execution initial expense, ease of control and insensitive to toxic substances. Because of its capability in recovery, regeneration and recycling of the adsorbent, made it known as one of the powerful and well-documented methods (Aljeboree et al., 2017). Activated carbon (AC) is porous carbon materials, possesses a large surface area (> 500 m^2/g), adsorption capability and adsorption capacity for gas to liquid phase application. It has been broadly applied for dye wastewater remedy as it did not demand any extra pre-treatment prior to its utilization (Abdus-Salam and Buhari, 2016). Yet, its costly expenditure in the market because of the cost of coal as precursor has restricted its economic utilization. Thus, curiosity in searching inexpensively, sustainable and readily obtainable materials as precursor has been executed, especially from agriculture wastes such as jute fibre, palm-tree cobs, olive stones, palm kernels, cassava peel, bagasse, date pits and fruit stones (Bedin et al., 2016). In this work, an endeavour was made by using lemon peel and mangosteen peel for the formation of AC.

1.3 Problem statement

The major problem faced by the textile industries is the removal of dyes from wastewater. The release of toxic effluents from different industries adversely influences soil fertility, aquatic organisms, water resources, and ecosystem integrity. Direct discharge of them had caused pollution of the water bodies. It is complex to forecast the attributes of textile wastewater by reported values in the literature as each industry is one of a kind in respect of the production, technology and chemicals used. The textile wastewater is stated as the most contaminating among all in the industrial sectors (Vilaseca et al., 2010; Awomeso et al., 2010). The textile industry utilizes an enormous amount of water and yield large amount of wastewater via different levels in dyeing and finishing processes. The textile wastewater consist of a multiple mixture of polluting matter like organic, inorganic, elemental and polymeric products (Charles et al., 2014). Among complicated industrial wastewater with different sorts of colouring agents, dye wastes are dominant. The textile wastewater consists of dye substances isn't just harmful to the biological environment, its dark colour hinders sunlight that leads to severe difficulty to the ecosystems (Nguyen et al., 2013).

Conventional furnace used during activation process heat the sample via conduction process. When the samples are exposed at high temperature for a long time (>1 hour), the energy consumption is high as well as less yield of AC was usually obtained. In this study, microwave heating is used as physiochemical activation which includes the use of potassium hydroxide, KOH and carbon dioxide, CO_2, as chemical agent and gasification agent, respectively which requires much lesser energy and time as compared to the conventional heating via furnace.

Among all the techniques, adsorption is one of the most capable techniques of eliminating dyes from waste sewage (Aljeboree et al., 2017). The process of adsorption has a benefit over the other technique because of its sludge free operation and perfect removal of dyes even from dilute solutions. AC had been completely made use of in different industrial adsorption and separation processes due to its capable adsorption of the organic compound. Nevertheless, owing to the high-priced of commercial coal based AC, its application in dye elimination from wastewater is restricted. Hence, this study was conducted to find out other alternatives precursor from agricultural waste which is cheap for preparing AC.

High demand for lemon and mangosteen fruits cause the plantation of these trees to increase, especially in southeast countries including Malaysia, Indonesia and Thailand. The part that are not eatable which is the lemon peel and mangosteen peel have limited application and most of the time, ended up as solid waste which required them to be properly managed and disposed. Global production of citrus fruit has significantly increased during the past few years and has reached 82 million tons in the years 2009–2010, of which lemons – commercially the most important citrus fruit accounts for about 50 million tons (USDA, 2010), and 34% of which was used for juice production, yielding about 44% peel as by-product. The edible aril of mangosteen is milky white, while the peel is dark red and composes about two times of the edible portion. Therefore, by converting lemon peel and mangosteen peel into AC, the cost to manage them as wastes can be eliminated and their potential as adsorbent can be exploited. Concerning to this, an attempt was made to use lemon peel and mangosteen peel wastes as precursor. The lemon peel based AC (LPAC) and mangosteen peel (MPAC) prepared were then tested its performance for adsorption of methylene blue (MB) and remazol brilliant blue R (RBBR) from aqueous solution.

1.4 Research objectives

The main purposes of this research are as follows:-

i. To prepare the optimized LPAC and MPAC preparation conditions (radiation power, activation time and impregnation ratio) by employing response surface methodology.
ii. To investigate the effect of methylene blue (MB) and remazol brilliant blue R (RBBR) adsorption onto LPAC and MPAC in batch process under various initial dye concentrations, solution temperature and contact time.
iii. To assess the adsorption isotherms, kinetics and thermodynamic attributes of MB and RBBR adsorption towards LPAC and MPAC.

1.5 Scope of study

In this research, the lemon peel and mangosteen peel were used to prepare AC for MB and RBBR dye removal. The provision of LPAC and MPAC were performed via physiochemical method that uses impregnation of KOH to improvise the adsorptive characteristic of the AC. The optimizations of the functional parameters of radiation power, activation time and impregnation ratio (IR) were done using response surface methodology (RSM) technique. RSM generates the structure of the experiment and the responses for each experimental run were analysed to obtain ideal operating conditions for the formulation of LPAC and MPAC for dye removal together with LPAC and MPAC yield.

The optimized LPAC and MPAC were later described with regard of surface area, surface morphology, surface chemistry and proximate content with the help of surface area analyzer, SEM, FTIR and STA respectively.

The optimized LPAC and MPAC were consequently used in equilibrium, kinetic and thermodynamic studies to look into the adsorption performance of each dye (MB and RBBR) onto LPAC and MPAC. In an effort to execute the analysis, batch adsorption study was executed by examining the effect of adsorbate initial concentration (25 – 300 mg/L), contact time (0 – 24 hour) and solution temperature (30 – 60 °C) for adsorption of dyes onto LPAC and MPAC prepared.

1.6 Thesis organization

This thesis contain of five major chapters and every chapter provides for the order of this study. The content of the chapters is given as follows:-

Chapter 1 presents the application of dyes in textile industries, problem statement, research objectives and thesis organization.

Chapter 2 discusses the literature review of this research. An understanding into dyes, discussion on adsorption process, activated carbon and raw material applied in developing activated carbon are elaborated. Furthermore, the isotherm models, kinetic models and thermodynamic parameters results are covered as well.

Chapter 3 covers the experimental materials and the details of the methodology. It discuss on the description of equipment and materials used, batch adsorption experiment, experimental procedure and description of factors affecting the adsorption process.

Chapter 4 refers to the experimental outcome and discussions of the data achieved. Additional discussion on the effect of different factors on batch system adsorption, the results in equilibrium, kinetic and thermodynamic properties are delivered in this chapter.

Chapter 5 summarizes all the information acquired in this research. Recommendations are added as well.

CHAPTER TWO

LITERATURE REVIEW

2.1 Dyes

Dyes are fundamentally chemical compounds that are able to attach themselves to surfaces or fabric to provide colour. The preponderance of dyes is complex organic molecules and are needed to be repellent to various items, for instance the response of detergents. Synthetic dyes are generally utilized in many fields of advanced technology, e.g., in different types of the textile (Abdus-Salam and Buhari, 2016), paper (Doumic et al., 2015), leather tanning (Charles et al., 2014), food processing, plastics, cosmetics, rubber, printing and dye fabrication industries (Dawood and Kanti Sen, 2014). Synthetic dyes are even well utilized in ground water tracing (Ismaiel et al., 2013), for the measurement of specific surface area of activated sludge (Li, Y. et al., 2016), sewage (Mouni et al., 2011) and wastewater treatment (Ahmed, 2016b), etc. Their release into the hydrosphere contain a substantial origin of contamination because of their recalcitrance nature. This will give unwanted colour to the water body which will decrease sunlight invasion and repel photochemical and biological assaults to aquatic life (Depci et al., 2012).

There are various techniques for categorizing of commercial dyes. They are separated with regard of structure, colour and application methods (Kyzas et al., 2012). Nevertheless, owing to the difficulty of the colour nomenclature from the chemical structure system, the categorization with respect to application is often favourable (Djilani et al., 2015). Table 2.1 presents the various usage based on categorization.