RESORCINOL – FORMALDEHYDE ACTIVATED CARBON GELS FOR ADSORPTION OF METHYLENE BLUE AND CONGO RED

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ABSTRACT

The issues related to the presence of inorganic and organic pollutants such as dyes in water bodies has become a subject of considerable concern. Chemically-formulated carbon gel is a promising material for continuous adsorption in wastewater treatment because it can be mould into desired shape to decrease hydraulic resistance leading to high pressure drop and operating costs. The effluents need a decisive treatment before been discharged to water resources. This work aims to evaluate the adsorption properties of activated carbons prepared from resorcinol-formaldehyde for the removal of positively charged methylene blue and negatively charged congo red from water. The activated carbons were prepared by chemical activation using potassium hydroxide at activation temperature of 550 °C for 2 h. The physicochemical properties of the activated carbons were characterized using Fourier transform infrared spectroscopy, thermal gravimetric analysis, Brunauer-Emmett-Teller (BET) surface area, pH value at the point of zero charge, Boehm titration, scanning electron microscope and elemental analysis. The BET surface area of the activated carbons are 427 and 433 m^2/g for resorcinol to catalyst ratios of 100 and 2000, respectively. The adsorption of methylene blue and congo red were studied at varying concentrations (10 – 300 mg/L) and contact times (1 h – 288 h). The isotherm and kinetics models were employed to describe the adsorption data. The maximum adsorption of methylene blue is 135 mg/g, while the maximum adsorption of congo red is 16 mg/g. The equilibrium data of methylene blue adsorption fitted well with the Freundlich and Redlich-Peterson models, while that of congo red adsorption obeyed Langmuir and Redlich-Peterson models. The kinetics data were best fitted to pseudo-first-order and pseudo-second-order models, indicating that both physisorption and chemisorption may have occurred simultaneously. The intraparticle diffusion model revealed that the intraparticle diffusion may be involved, but it is not the only rate – limiting step, while Boyd model showed that film diffusion may be the controlling mechanism. For adsorption dynamic, the adsorption of methylene blue was studied at varying bed heights (2 and 6 cm), flow rates (5, 10 and 20 mL/min) and inlet concentrations (5, 10 and 20 mg/L). The results show that the performance of activated carbons for methylene blue adsorption is better at lower flow rate, lower inlet concentration and higher activated carbon bed height. The adsorption dynamic data of methylene blue adsorption fitted well with Thomas, Yoon-Nelson and Adams-Bohart, indicating that the kinetics system was controlled by external mass transfer or film diffusion.

ABSTRAK

Isu yang berkaitan dengan kehadiran bahan cemar inorganik dan organik seperti pencelup dalam jasad air sudah menjadi perkara yang membimbangkan. Gel karbon yang diformulasikan secara kimia merupakan bahan yang sesuai untuk penjerapan secara berterusan dalam rawatan air kumbahan kerana ia boleh diacu kedalam bentuk yang sesuai bagi mengurangkan rintangan hidraulik yang membawa kepada penurunan tekanan dan kos operasi yang tinggi. Oleh sebab itu, cairan buangan memerlukan penentuan rawatan sebelum ia dilepaskan ke sumber air. Kajian ini bertujuan untuk menilai sifat – sifat penjerapan bagi karbon teraktif yang dihasilkan daripada resorsinol-formaldehid untuk penyingkiran metilena biru bercas positif dan kongo merah bercas negatif daripada air. Karbon teraktif dihasilkan oleh pengaktifan kimia menggunakan kalium hidroksida pada suhu pengaktifan iaitu 550 °C selama 2 jam. Sifat fizikokimia karbon teraktif telah dicirikan menggunakan spektroskopi inframerah jelmaan Fourier, analisa terma gravimetrik, luas permukaan Brunauer-Emmett-Teller (BET), nilai pH pada titik sifar cas, pentitratan Boehm, mikroskop imbasan eletron dan analisis unsur. Luas permukaan BET bagi karbon teraktif adalah masing-masing 427 dan 433 m^2/g bagi nisbah resorsinol kepada mangkin 100 dan 2000. Penjerapan metilena biru dan merah kongo telah dikaji pada pelbagai kepekatan (10 – 300 mg/L) dan masa sentuh (1 jam – 288 jam). Model isotherma dan kinetik telah digunakan untuk menerangkan data penjerapan. Penjerapan maksimum bagi metilena biru ialah 135 mg/g manakala penjerapan maksimum bagi merah kongo ialah 16 mg/g. Data keseimbangan bagi penjerapan metilena biru sesuai dengan model Freundlich dan Redlich-Peterson, manakala penjerapan merah kongo mematuhi model Langmuir dan Redlich-Peterson. Data kinetik adalah sangat sesuai dengan model tertib pseudo-pertama dan tertib pseudo-kedua yang mana menunjukkan kedua – dua penjerapan fizikal dan penjerapan kimia mungkin berlaku secara serentak. Model resapan intrazarah menunjukkan bahawa resapan intrazarah mungkin berlaku, tetapi ia bukan langkah kadar-penghad tunggal, manakala model Boyd menunjukkan bahawa resapan filem mungkin merupakan mekanisma kawalan. Bagi penjerapan dinamik, penjerapan bagi metilena biru telah dikaji pada pelbagai ketinggian padatan (2 dan 6 cm), kadar aliran (5, 10 dan 20 mL/min) dan kepekatan salur masuk (5, 10 dan 20 mg/L). Keputusan menunjukkan bahawa penjerapan bagi metilena biru beraksi dengan baik pada kadar aliran yang rendah, kepekatan salur masuk yang rendah dan tinggi ketinggian padatan karbon teraktif. Data penjerapan dinamik bagi penjerapan metilena biru bersesuaian dengan Thomas, Yoon-Nelson dan Adams-Bohart, yang mana menunjukkan sistem kinetik telah dikawal oleh perpindahan jisim secara luaran atau resapan filem.

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LIST OF ABBREVIATIONS

MB	-	Metyhlene Blue
CR	-	Congo Red
AC	-	Activated Carbon
KOH	-	Potassium Hydroxide
Na_2CO_3	-	Sodium Carbonate
CO_2	-	Carbon Dioxide
RF	-	Resorcinol - Formaldehyde
H_3PO_4	-	Phosphoric Acid
$ZnCl_2$	-	Zinc Chloride
NaOH	-	Sodium Hydroxide
$FeCl_2$	-	Iron Chloride
K_2CO_3	-	Potassium Carbonate
HCl	-	Hydrochloric Acid
NiO	-	Nickel Oxide
TBA	-	Tert-Butyl-Alcohol
RF-AC	-	Resorcinol – Formaldehyde Activated Carbon
KBr	-	Potassium Bromide
FT-IR	-	Fourier Transform Infrared Spectroscopy
$NaHCO_3$	-	Sodium Bicarbonate
BET	-	Brenauere-Emmett-Teller
TGA	-	Thermogravimetric Analysis
pH_{PZC}	-	pH of The Point of Zero Charge
SEM	-	Scanning Electron Microscopy
CH_4	-	Methane
R/C	-	Resorcinol to Catalyst Ratio
Uv-Vis	-	Ultraviolet-Visible Spectroscopy
DTG	-	Derivative Thermogravimetry
N_2	-	Nitrogen

LIST OF SYMBOLS

°C	-	Degree Celcius
m^2	-	Meter Square
g	-	Gram
mg	-	Milligram
p	-	Pressure
L	-	Litre
%	-	Percent
ppm	-	Parts Per Million
h	-	Hour
min	-	Minute
mL	-	Millilitre
mmol	-	Millimoles
µm	-	Micrometer
cm	-	Centimeter
λ_{max}	-	Maximum Wavelength
q_e	-	Amount of Adsorbate Adsorbed per Unit Mass of Adsorbent at Equlibrium
C_o	-	Initial Dye Concentration
C_e	-	Equilibrium Dye Concentration
b	-	Langmuir Isotherm Constant
Q_m	-	Maximum Monolayer Coverage Capacities
K_F	-	Freundlich Isotherm Constant
W	-	Mass of Activated Carbon
n	-	Adsorption Intensity
K_R	-	Redlich-Peterson Isotherm Constant 1
α_R	-	Redlich-Peterson Isotherm Constant 2
nm	-	Nanometer
pKa	-	Acid Strength
β	-	Desorption Constant
k_1	-	Rate Constant for Pseudo-First-Order Adsorption

k_2	-	Rate Constant for Pseudo-Second-Order Adsorption
k_d	-	Diffusion Coefficient for Intraparticle Diffusion
S_{BET}	-	BET Surface Area
K_{ad}	-	Dubinin-Radushkevich Isotherm Constant
E	-	Mean Free Energy of Adsorption
R_L	-	Separation Factor
K_{TH}	-	Thomas Rate Constant
N_o	-	Saturation Concentration
K_{YN}	-	Yoon-Nelson Rate Constant
τ	-	Time for 50 % Breakthrough
K_{AB}	-	Adams-Bohart Rate Constant

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The issues that are related on the release of inorganic and organic pollutants into the water bodies have become a subject of considerable concern. The waste water contains toxic substances such as heavy metals, organic chemicals and dyestuffs exceeding the maximum permissible limit set by the local authorities (WHO, 2006). One of the major wastewater effluent is dye. There are various type of dyes were discharge from the industries such as acid dye, basic dye, direct dye and azoic dye. In this research, methylene blue (MB) (3,7-bis(Dimethylamino)-phenothiazin-5-ium chloride) and congo red (CR) [1-naphthalene sulfonic acid, 3,30-(4,40-biphenylenebis (azo)) bis(4-amino-) disodium salt] have been chosen as the model pollutants because they are commonly used in textile industries.

The damaging effect of water contaminants gives impacts not only to the aquatic ecosystem but also to human who depends on water resources for routine life. Due to that, the effluents need a decisive treatment before it pollutes the water resources. Among all of the extensive strategies of treatment, adsorption has appeared as the widely tested technique in wastewater treatment. Adsorption is a process by which the contaminants (solutes) are adhered to the solid surface of the adsorbent through numerous mechanisms for examples л-л interaction, ion exchange, pore filling, and the formation of complex or chelate (Ming-Twang *et al.*, 2015).

In addition, adsorption through continuous mode is more suitable for industry that involves a massive volume of wastewater due to simple and easy operation, less space needed and also minimum costs of manufacturing. However, many of commercial adsorbents available today are in the powder and granular forms which unfit for continuous process. In spite of large specific surface area and rich surface

chemistry for satisfactory removal performance, the column operation may suffer in escalating pressure drop caused by the hydraulic resistance if adsorbent with tiny particles is employed (Masuda *et al.*, 2013). As a result, the column performance will become poor and the operating costs may be high.

A potential solution for this impediment is a packing – type adsorbent called carbon gel. Carbon gel can be formed through polymerization of resorcinol and formaldehyde in the presence of solvent (water) and basic catalyst (sodium carbonate) (Rojas-Cervantes, 2015). The mesoporous carbon structure is established via the aggregation of nanometer-sized colloidal particles. The formation of gaps between the nanoparticles developed a hierarchical pore system of mainly mesopores, and some micropores and macropores (Mukai, 2012). Carbon gel is a porous material with large carbon density that can be shaped into desired size and customized for definitive utilizations. For examples, catalysts for chemical synthesis (Mukai, 2012), adsorbents for chemical separation and environmental protection (Al-Muhtaseb & Ritter, 2003), and also as carbon electrodes for energy storage (Tsuchiya *et al.*, 2014). Due to these exclusive quality, carbon gel become a promising candidate to overcome the resistance of flow and provide a short path for efficient connection between the solute and solid surface in continuous system of adsorption (Masuda *et al.*, 2013).

Nowadays, the application of carbon gel as adsorbent for the removal of pollutants especially in continuous adsorption is not widely available in literature. This could be due to its low adsorption performance. Therefore, the primary objective of this research is to synthesize and characterize activated carbon gels using resorcinol-formaldehyde sol-gel method, followed by chemical activation using potassium hydroxide (KOH). The chemical activation of carbon gels using KOH is expected to enhance the adsorption performance of materials. This study was performed to evaluate the performance of activated carbon gels in the removal of methylene blue and congo red and also the column performance from the viewpoints of concentration, flow rate, and bed height.

1.2 Problem Statement

Commercial and laboratory-made activated carbons are mainly in granular and powdered forms. The small particles size of activated carbon often results in hydraulic resistance leading to escalating pressure drop in column adsorption. Hence, decreasing the throughput and increasing the operating costs. Chemically-formulated carbon gel can be mould into desired shape/dimension, hence a promising material for continuous adsorption. It can be produced using sol-gel method of resorcinol-formaldehyde in the presence of sodium carbonate catalyst. However, the adsorption of water pollutants in continuous mode is not widely documented in literature. This could be due to its low adsorption performance. Activation using potassium hydroxide (KOH) that is normally performed at lower activation temperature can produce adsorbent with improved adsorptive properties. Therefore, attempt has been made in this study to activate resorcinol-formaldehyde carbon gel for the removal of methylene blue and congo red. The study involved the characterization of activated carbon gels, the assessment of performance by activated carbon gels in the removal of model water pollutants, namely methylene blue and congo red, and also the column performance.

1.3 Objective of Research

The aims of the study are:

(a) To synthesize and characterize activated carbon gels derived from resorcinol – formaldehyde by potassium hydroxide activation.

(b) To evaluate the performance of activated carbon gels in the removal of methylene blue and congo red by using batch and continuous modes of adsorption.

(c) To evaluate the adsorption mechanisms of dyes according to isotherm, kinetics and dynamics models.

1.4 Scope of Research

Carbon gel was synthesized using resorcinol and formaldehyde with the present of sodium carbonate which act as catalyst. The ratios of resorcinol to catalyst used were 100 and 2000. The carbon gels were chemically activated using potassium hydroxide at a KOH : carbon gel ratio of 2:1 at 550 ºC for 2 h. Then, the activated carbon gels were characterized using Fourier transform infrared (FT-IR) spectroscopy, Brunauer–Emmett–Teller (BET) surface area analyzer, thermogravimetric analysis (TGA), scanning electron microscope (SEM), pH_{PZC}, CHNS analyzer and Boehm titration.

The performance of activated carbon gels in batch mode were evaluated by the removal of methylene blue and congo red at different concentrations (10 to 300 mg/L) and contact times (1 h to 288 h). Column adsorption was used to evaluate the continuous process at different flow rates, bed heights and inlet concentrations. The flow rates are 5, 10 and 20 mL/min, while the bed heights of carbon gels are 2 and 6 cm. The concentration of dyes are varied at 5, 10 and 20 mg/L.

1.5 Significance of Research

This study was carried out to give further comprehension on the wastewater treatment process of water pollution using activated carbon gel in batch and column modes. Adsorption process is one of the favoured method for the removal of water pollutant due to simple, cost-effective and dynamic operation, and no production of sludge. In this research, activated carbon gels were used to evaluate the removal of methylene blue and congo red. The materials were made up from resorcinol and formaldehyde with the present of basic catalyst, followed by KOH activation. This research is expected to shed some light on the column performance of activated carbon gels from the aspect of inlet concentration, bed height and flow rate.

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