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REMOVAL OF DYES USING CITRIC ACID MODIFIED OIL PALM SHELL

By

YUEN MEI LIAN

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

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DEDICATION

To my beloved parents Yuen Poo Haak and Sam Guan Hah, and my siblings

For their endless love, support and concern

To my supervisor Prof. Dr. Zulkarnain Bin Zainal

For his valuable guidance, understanding and advices

To my co-supervisor Assoc. Prof. Dr. Abdul Halim Bin Abdullah

For his advices, encouragements and supports

To my seniors Dr. Lee Kong Hui, Mr. Lee Chong Yong and Ms. Chang Sook Keng

For their wonderful advices and supports

To my friends

For their moral supports and encouragement

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

REMOVAL OF DYES USING CITRIC ACID MODIFIED OIL PALM SHELL

By

YUEN MEI LIAN

October 2009

Chairman: **Professor Zulkarnain Zainal, PhD**

Faculty: **Science**

This study investigates the effectiveness of using modified oil palm shell (MPS) to remove dye from solution. The feasibility of modified oil palm shell in removing basic dyes from solution was evaluated and compared with the unmodified oil palm shell (NPS). The optimized modification condition was obtained by treating 1.00 g of oil palm shell with 7 mL of 1.20 M citric acid in an oven at 120 °C for 90 minutes. The prepared adsorbent was characterized by a Scanning Electron Microscopy and Fourier Transform Infrared Spectroscopy.

In each adsorption experiments, 200 mL of dye solution of known concentration and pH was added to a certain amount of adsorbent and the mixture was stirred for 4 hours. Batch studies were performed under various experimental conditions and the parameters studied include initial pH of solution, contact time on different initial dye concentrations, temperature, particle size and adsorbent dosage. The desorptibility of

the adsorbent and its ability to remove other dyes were also examined. Besides that, isotherm studies were done to determine the adsorption capacity of NPS/MB, MPS/MB and MB-loaded MPS/IC systems by varying the dye concentration solutions.

In the MPS/MB system, the adsorption of MB by MPS was pH-dependent. The maximum MB removal found at pH 11 was 58%. By increasing the temperature from 28 to 40 ºC of MB solution (pH 5.3), the removal efficiency has been enhanced from 66 to 72%, indicated adsorption process was endothermic. Reducing adsorbent particle size from 500 to 106 µm led to an increase in the adsorption of MB from 12 to 52% due to more available surface area. Similar finding was also observed in the increment of adsorbent dosage (from 0.10 to 0.40 g). The desorption process was unfavourable either in acidic or basic condition. The highest percentage of desorption was only 13% at pH 0.

The highest adsorption percentage for 50 ppm MB by NPS and MPS was 81% and 94%, respectively. The kinetics of MB adsorption in both systems can be better fitted to a pseudo-second order rate expression than pseudo-first order rate expression. The Langmuir equation gave a better fit to the NPS/MB and MPS/MB systems than Freundlich equation. The adsorption capacity of MB onto MPS, 73.53 mg/g, was higher compared to NPS, 44.05 mg/g, suggested MPS was a better adsorbent for the removal of MB from aqueous solution.

In the removal of other dyes, MPS appeared to be a poor adsorbent for the removal of 10 ppm anionic dyes (Methyl Orange, Indigo Carmine and Reactive Blue 2), which is less than 7%. Meanwhile, adsorption capacity towards anionic dyes has changed significantly up to 65% by using MB-loaded MPS due to the electrostatic attraction. In MB-loaded MPS/IC system, kinetic data of adsorption was well fitted to the pseudo-second order kinetic model. It has been established that the adsorption of IC on MB-loaded MPS followed the Langmuir isotherm equation and the maximum removal capacity was 8.76 mg/g.

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

PENYINGKIRAN PEWARNA DENGAN MENGGUNAKAN TEMPURUNG KELAPA SAWIT YANG SITRIK-UBAHSUAIAN

Oleh

YUEN MEI LIAN

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Penyelidikan ini mengkaji keberkesanan menggunakan tempurung kelapa sawit terubahsuai (MPS) untuk menyingkirkan bahan pewarna daripada larutan. Kebolehan MPS dalam penyingkiran bahan pewarna basik daripada larutan dinilai dan dibandingkan dengan tempurung kelapa sawit tidak terubahsuai (NPS). Keadaan optimum untuk modifikasi telah didapati dengan menindakbalaskan 1.00 g NPS dengan 7 mL 1.20 M asid sitrik di dalam ketuhar pada suhu 120 °C selama 90 minit. Pencirian bahan penjerap yang disediakan dilakukan melalui Mikroskopi Pengimbasan Elektron dan Spektroskopi Inframerah Transformasi Fourier.

Dalam setiap eksperimen jerapan, kepekatan dan pH bagi 200 mL larutan pewarna yang diketahui ditambah masuk dengan amaun bahan penjerap tertentu dan campuran tersebut dikacau selama 4 jam. Kajian kelompok telah dijalankan di bawah pelbagai keadaan eksperimen dan parameter yang dikaji termasuklah pH awal larutan,

kepekatan awal pewarna, suhu, saiz partikel dan dos bahan penjerap. Kebolehan penyahjerapan bagi bahan penjerap dan keupayaan bahan penjerap untuk menyingkirkan pelbagai pewarna juga diuji. Selain itu, kajian isoterma telah dijalankan untuk menentukan kapasiti jerapan bagi sistem NPS/MB, MPS/MB dan MPS terdokong MB/IC dengan mempelbagaikan kepekatan larutan pewarna.

Dalam sistem MPS/MB, jerapan MB oleh MPS dipengaruhi oleh pH. Peratusan penyingkiran yang maksimum dapat diperhatikan pada pH 11 ialah 58%. Dengan meningkatkan suhu larutan MB (pH 5.3) dari 28 ke 40 ºC, tahap penyingkiran dapat ditingkatkan dari 66 ke 72%, menunjukkan proses jerapan adalah endotermik. Pengurangan saiz partikel bahan penjerap dari 500 ke 106 µm menghasilkan peningkatan jerapan MB dari 12 ke 52% disebabkan terdapat luas permukaan yang lebih banyak. Pemerhatian yang sama juga diperhatikan dalam peningkatan dos bahan penjerap (dari 0.10 ke 0.40 g). Proses penyahjerapan tidak cenderung sama ada dalam keadaan asid atau basik. Peratusan penyahjerapan adalah hanya 13% pada pH 0.

Peratusan jerapan tertinggi untuk 50 ppm MB oleh NPS dan MPS ialah 81% dan 94%. Kinetik bagi jerapan MB bagi kedua-dua sistem adalah lebih sesuai mengikut hukum kadar pseudo-kedua berbanding hukum kadar pseudo-pertama. Persamaan Langmuir merupakan isoterma jerapan yang lebih sesuai bagi sistem NPS/MB dan MPS/MB. Kapasiti jerapan MB pada MPS ialah 73.53 mg/g, lebih tinggi berbanding dengan pada NPS, 44.05 mg/g, mencadangkan MPS merupakan bahan penjerap yang lebih baik bagi penyingkiran MB dari larutan akueous.

Dalam penyingkiran pewarna lain, MPS menunjukkan bahan penjerap yang lemah dalam penyingkiran pewarna anionik (Metil Oren, Indigo Karmin dan Reaktif Biru 2), iaitu kurang daripada 7%. Namun begitu, kapasiti jerapan terhadap pewarna anionik telah bertambah secara mendadak sehingga 65% dengan sistem MB terdokong MPS/IC disebabkan tarikan elektrostatik. Dalam sistem MPS terdokong MB/IC, data kinetik jerapan sekali lagi didapati sesuai menggunakan model kinetik hukum kadar pseudo-kedua. Jerapan IC oleh MPS terdokong MB mengikut persamaan isoterma Langmuir dengaan kapasiti jerapan maksimum 8.76 mg/g.

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I certify that a Thesis Examination Committee has met on 13 October 2009 to conduct the final examination of Yuen Mei Lian on her thesis entitled "**Removal of Dyes Using Citric Acid Modified Oil Palm Shell**" 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 degree of Master of Science.

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Date: 11 February 2010

DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

____

YUEN MEI LIAN

Date:

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LIST OF ABBREVIATIONS

AG25	Acid Green 25
AO10	Acid Orange 10
AB10B	Amino Black 10B
b	Langmuir constant
BB3	Basic Blue 3
C	Intercept
C_a	Concentrations of dye adsorbed
C_d	Concentrations of dye desorbed
C_e	Equilibrium concentration of dye
C_o	Initial concentration of dye
FTIR	Fourier Transform Infrared
h	Initial sorption rate
IC	Indigo Carmine
K_f	Freundlich isotherm constants for adsorption capacity
k_1	Rate constant of pseudo-first order adsorption
k_2	Rate constant of pseudo-second order adsorption
k_i	Rate constant of intraparticle diffusion model
MB	Methylene Blue
MO	Methyl Orange
MPS	Modified oil palm shell
n	Freundlich isotherm constants for adsorption intensity
NPS	Unmodified oil palm shell

q^*	Langmuir constants related to maximum adsorption capacity
q_e	Amount of dye adsorbed at equilibrium
q_i	Amount of dye adsorbed at time t for intraparticle diffusion model
q_t	Amount of dye adsorbed at time t
R^2	Correlation coefficient
R_L	Dimensionless constant
RB2	Reactive Blue 2
RO16	Reactive Orange 16
RR141	Reactive Red 141
SEM	Scanning Electron Microscopy
T	Temperature
t	Time
$t^{1/2}$	Values of half time
UV/Vis	Ultra violet/visible
WBA	Wood-shaving bottom ash
λ_{max}	Wavelength