UNIVERSITI PUTRA MALAYSIA

PREPARATION, CHARACTERIZATION AND METHYLENE BLUE ADSORPTION OF SURFACTANT-TEMPLATED MESOPOROUS CARBON COATED MONOLITH

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MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA

2012

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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By

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AUGUST 2012

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Faculty : Engineering

This thesis work brings the preparation and characterization of mesoporous carbon coated monolith (MCCM) by using nonionic surfactant as template and its application on adsorption of methylene blue. MCCM with high surface area and pore volume was synthesized and characterized in detail. The preparation of mesoporous carbon coated monoliths consisted of polymerization, dip-coating, solidification and carbonization. MCCM have been prepared by dip-coating cordierite monoliths in a polymer solution consists of furfuryl alcohol (carbon source), F127 (template), pyrrole (binder) and nitric acid (polymerization catalyst). After solidification of the polymer, the coating was carbonized at temperature of 700 ^{o}C to obtain MCCM. The influence of concentration of the surfactant template in MCCM was investigated. It was found that by increasing the concentration of surfactant, adsorption capacity of MCCM increases from 100 to 200 mg/g. The mesoporous carbon coated monoliths are characterized by nitrogen adsorption-desorption, thermogeravimetric analysis, scanning electron microscopy and Fourier transform infrared. The characterization study shows that the MCCM prepared in

this work possesses a higher surface area (842 m^2/g), pore volume (0.3 m^3/g) and mesoporosity (97%) compare to previous study (MCCM by PEG). The potential of mesoporous carbon coated monolith on the adsorption of methylene blue dye was investigated. Batch adsorption experiments were carried out to find out the effects of pH (2-11), salt, contact times (4480 min), initial dye concentrations (50-400 ppm) and temperature (25, 35 and 45 ^oC) on the adsorption of the dye. pH was found to be the most significant factor and pH 10 was favorable for MB adsorption. Furthermore, kinetic study showed that the adsorption could be better represented by the pseudo-second-order model. Equilibrium adsorption data were predicted by two isotherms, i.e. the Langmuir and the Freundlich isotherms. The best fit to the data was obtained with the Langmuir isotherm with maximum monolayer adsorption capacity of 388 mg/g. Adsorption of MB is favourably influenced by an increase in the temperature of the operation from 388 to 440 mg/g. By associated thermodynamic parameters, the adsorption was found to be spontaneous and endothermic. Desorption study indicates that 0.1N hydrochloric acid exhibits higher elution efficiency and a quantitative recovery of MB (around 82%) can be achieved.

Abstrak tesis ini dikemukakan kepada Senat Universiti Putra Malaysia bagi memenuhi keperluan untuk ijazah Master Sains

PENYEDIAAN, PENCIRIAN, DAN PENJERAPAN METILENA BIRU KE ATAS MESOPOROUS MONOLIT BERSALUT KARBON DENGAN MENGGUNAKAN SURFAKTAN SEBAGAI TEMPLATE

Oleh

MOHAMAD RASOOL MALEKBALA

OGOS 2012

Pengerusi : Profesor Thomas Choong Shean Yaw, PhD

Fakulti : Kejuruteraan

Tesis ini merangkumi penyediaan dan pencirian karbon mesoporous bersalut monolit (MCCM) dengan menggunakan surfaktan tidak berion sebagai acuan dan aplikasinya dalam penjerapan metil etena biru. MCCM yang mempunyai luas permukaan dan isipadu liang yang tinggi dihasilkan dan dicirikan dengan teliti. Penyediaan mesoporous monolit bersalut karbon adalah melalui kaedah pempolimeran, celup dan salut, pemejalan dan karbonisasi. MCCM disediakan dengan mencelupdan salut kordierit monolit ke dalam larutan polimer yang mengandungi alkohol furfuryl (sumber karbon), F127 (bahan acuan), pirola (ikatan) dan asid nitrik (pemangkin pempolimeran). Setelah pemejalan polimer dilakukan, salutan tadi dikarbonkan pada suhu 700 ^{o}C untuk menghasilkan MCCM. Kajian mengenai pengaruh kepekatan surfaktan di atas acuan di dalam MCCM telah dikaji. Hasil analisis menunjukkan bahawa dengan meningkatkan kuantiti surfaktan, keupayaan penjerapan MCCM turut meningkat dari 100 ke 200 mg/g. Mesoporous monolit bersalut karbon telah dicirikan melalui proses penjerapan dan penyahjerapan

nitrogen, analisis geravimetrik termo, analisis pengimbasan mikroskop elektron dan juga melalui kaedah Fourier inframerah. Hasil kajian menunjukkan bahawa penyediaan MCCM melalui kaedah ini telah berupaya untuk menghasilkan luas permukaan dan liang isipadu yang tinggi berbanding dengan kajian terdahulu (MCCM menggunakan PEG). Potensi mesoporous karbon yang bersalut monolit terhadap penjerapan pewarna metil etena biru telah dikaji. Eksperimen penjerapan secara berkumpulan telah dijalankan untuk melihat kesan terhadap pH (2-11), garam, masa sentuh (4480 min), kepekatan asal pewarna (50-400 ppm) dan suhu (25, 35 and 45 ^{o}C) ke atas penjerapan warna. Kesan pH didapati menunjukkan keputusan amat signifikasi dan pH 10 adalah terbaik untuk penjerapan MB. Tambahan pula, kajian kinetik menunjukkan bahawa penjerapan adalah lebih baik dengan menggunakan model pseudo-tertib-kedua. Keseimbangan penjerapan data telah diramal dengan menggunakan dua isoterma iaitu Langmuir dan Freundlich. Padanan terbaik data telah diperolehi melalui isoterma Langmuir dengan keupayaan penjerapan maksimum sebanyak 388 mg/g. Penjerapan MB banyak dipengaruhi oleh peningkatan suhu kajian dari 388 ke 400 mg/g. Penjerapan dilihat sebagai spontan dan endotermik berpandukan kepada termodinamik parameter . Penyahjerapan menunjukkan bahawa penggunaan 0.1N asid hidroklorik boleh mempamerkan kecekapan elusi yang lebih tinggi dan pemulihan kuantitatif MB (anggaran 82%) dapat dicapai.

ACKNOWLEDGEMENTS

In the name of Allah the Most Merciful and Most Beneficent, to whom I express all the thankfulness and gratitude as if not of HIM I would not able to finish this work. Without HIM I lacked the knowledge, strength and patience to overcome the tough and unpredicted obstacles I had faced along this journey. I shall be thankful to HIM all my life, Insya-Allah.

I wish to express my sincere gratitude and appreciation to my supervisor Associate Professor Thomas Choong Shean Yaw for his comments and personal guidance throughout my study. I also wish to thank my co-supervisors Professor Luqman Chuh Abdullah and Dr. Soraya Hosseini for their valuable input to this work.

I am very much gratified to all the academic staff, technicians and administrative staff of the Department of Chemical and Environmental Engineering. My thanks are also extended to all my friends and colleagues who gave me all kind of support during my study.

I would like to express my most sincere and warmest gratitude to my parents, brothers, and sisters for their prayer, loving, and moral inputs especially during the most difficult times I faced along this period.

I certify that an Examination Committee has met on 28th of August 2012 to conductthe final examination of name of Mohamad Rasool Malekbala on his Master of Science thesis entitled "Preparation, Characterization and Methylene Blue Adsorption of Surfactant-Templated Mesoporous Carbon Coated Monolith" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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This thesis was submitted to the Senate of 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

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 it is not concurrently, submitted for any other degree at Universiti Putra Malaysia or other institutions.

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MOHAMAD RASOOL MALEKBALA

Date: 28 August 2012

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