UNIVERSITI PUTRA MALAYSIA

PREPARATION OF FURFURYL ALCOHOL-DERIVED ACTIVATED CARBON MONOLITH FOR LIQUID ADSORPTION

DARMADI

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By

DARMADI

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PREPARATION OF FURFURYL ALCOHOL DERIVED-ACTIVATED CARBON MONOLITH FOR LIQUID ADSORPTION

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DARMADI

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Faculty : Engineering

The preparation and optimization of carbon coated monolith is reported. The aim is to produce mesoporous activated carbon monolith for liquid adsorption using the dipcoating method. The materials required are a carbon source (furfuryl alcohol), a pore former agent (poly ethylene glycol), a binder (pyrrole), and polymerization catalyst (nitric acid). Furfuryl alcohol (FA) is first polymerized, followed by the impregnation of monolithic structure, carbonization, and activation. The effect of poly ethylene glycol (PEG) on the structure of carbon monolith is first investigated. The carbon coated monoliths are characterized by thermo gravimetrical analysis (TGA), elemental analysis, scanning electron microscopy (SEM), Fourier transform infrared (FTIR) and textural analysis. The carbon monolith prepared without the addition of pore former agent (only FA) exhibits adsorption Type I which is a characteristic of microporous material, whilst the carbon monolith prepared with the addition of pore former agent (FA +

PEG) is of Type IV indicating mesoporous material. Brunauer, Emmett, and Teller (BET) surface areas measured by N_2 adsorption are 264 and 431 $m^2 g^{-1}$ for sample FA and sample (FA + PEG), respectively. Total pore volume of the samples FA and FA + PEG are 0.13 and 0.38 $cm^3 g^{-1}$, respectively. The PEG is completely decomposed during carbonization to create new mesoporosity.

The optimization of pore volume and surface area of carbon coated monolith is studied using the response surface methodology (RSM) based on the Box-Behnken design. The carbonization temperature, concentration of PEG, and molecular weight of PEG are identified as the dominant parameters in controlling the pore size distribution, pore volume, and surface area. The maximum pore volume found from the RSM is 173 $mm^3 g^{-1}$ at carbonization temperature of 680 ^oC and concentration of PEG of 38% vol. with molecular weight of PEG of 1000 g mol^{-1}, whilst maximum surface area is 585 $m^2 g^{-1}$ at carbonization temperature of 660 ^oC and concentration of PEG of 31% vol. with molecular weight of PEG of 1000 g mol^{-1}. To confirm these results, synthesis of carbon coated monolith is performed. Experimental results obtained are pore volume of 161 $mm^3 g^{-1}$ and surface area of 553 $m^2 g^{-1}$, which are very close to the prediction by RSM.

The performance of activated carbon monolith is evaluated using the methylene blue (MB) adsorption. Equilibrium adsorption data are predicted by three isotherms, i.e. the Langmuir, the Freundlich, and the Redlich-Peterson isotherms. The best fit to the data is obtained with the Redlich-Peterson and the Langmuir isotherms with correlation coefficient (R^2) of 0.997 and 0.998, respectively. The maximum monolayer adsorption capacity is 191 mg g^{-1}. The Langmuir isotherm is used for modeling and simulation as it is a two parameter model and has similar accuracy in describing the isotherm data in this work. The dimensionless equilibrium parameter (R_L) is calculated as 0.1, indicating that the adsorption is favorable.

The kinetics of adsorption of MB is studied in terms of pseudo first order and second order mechanism for chemical adsorption as well as an intraparticle diffusion mechanism by applying the linear driving force (LDF) approximation in batch system. Kinetic parameters and correlation coefficients are determined. It is shown that the pseudo first order kinetic equation fits well to describe the adsorption kinetics with rate constants 6.6×10^{-3} min^{-1}, 4.0×10^{-3} min^{-1}, and 1.5×10^{-3} min^{-1} for initial concentrations 20, 50, and 100 mg L^{-1}, respectively.

The LDF model for a monolithic system is developed. The pseudo first order rate constant is used as initial guess to estimate the LDF mass transfer coefficient (k_{LDF}) by matching the simulation with experimental data. The comparison of the results calculated using the LDF model and experimental data is in good agreement with k_{LDF} values 8.25×10^{-3}, 5.20×10^{-3}, and 2.50×10^{-3} min^{-1} for initial concentrations 20, 50, and 100 mg/L, respectively.

A predictive dispersed plug flow model, with adsorption rate described by the LDF model, is developed to predict the breakthrough curve of a monolith column. The model parameters are from batch adsorption experiments. The result of simulation is found to agree excellently with the experimental data. The effect of LDF mass transfer coefficients is investigated numerically. The LDF mass transfer coefficients are found to significantly affect the shape of the breakthrough curve.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai keperluan untuk ijazah Doktor Falsafah

PENYEDIAAN MONOLIT BERKARBON TERAKTIF DARI *FURFURYL ALCOHOL* UNTUK PENJERAPAN

Oleh

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Oktober 2009

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Kajian penyediaan dan pengoptimuman monolit bersalut karbon telah dijalankan. Penyelidikan bertujuan untuk menghasilkan monolit berkarbon teraktif dengan liang bersaiz meso dengan kaedah salutan celup untuk penjerapan cecair. Bahan-bahan yang diperlukan adalah sumber karbon (*furfuryl alcohol*), agen pembentuk liang (PEG), pengikat (*pyrrole*), dan pemangkin pempolimeran (asid nitrik). *Furfuryl alcohol* (FA) dipolimerkan terlebih dahulu, kemudian diikuti dengan celupan dari struktur monolit, pengkarbonan, dan pengaktifan. Kesan agen pembentuk liang (PEG) terhadap struktur karbon monolit diselidik. Monolit bersalut karbon dicirikan menggunakan analisis termo gravimetrik (TGA), analisis berunsur, imbasan elektron mikroskopi (SEM), pemindahan Fourier inframerah (FTIR) dan analisis tekstur. Karbon monolit yang disediakan tanpa penambahan agen pembentuk liang (hanya FA) menunjukkan penjerapan Jenis I yang berciri bahan berliang mikro, manakala karbon monolit yang disediakan dengan

penambahan agen pembentuk liang (FA + PEG) adalah Jenis IV yang menunjukkan bahan berliang meso. Luas permukaan Brunauer, Emmett, dan Teller (BET) yang diukur menggunakan penjerapan N_2 adalah masing-masing 264 dan 431 m^2 g^{-1} untuk sampel FA dan sampel (FA + PEG). Manakala jumlah isipadu liang bagi sampel-sampel FA dan FA + PEG adalah masing-masing 0.13 dan 0.38 cm^3 g^{-1}. PEG terurai sepenuhnya semasa pengkarbonan untuk membentuk keliangan meso yang baru.

Penghasilan isipadu liang dan luas permukaan yang optimum bagi monolit bersalut karbon diselidik menggunakan *response surface methodology* (RSM) berdasarkan rekabentuk Box-Behnken. Suhu pengkarbonan, kepekatan PEG, dan berat molekul PEG dikenalpasti sebagai parameter yang dominan dalam pengawalan pengagihan saiz liang, isipadu liang, dan luas permukaan. Isipadu liang maksimum yang dikenalpasti daripada RSM adalah 173 mm^3 g^{-1} pada suhu pengkarbonan 680 ^oC dan kepekatan PEG berisipadu 38% dengan berat molekul PEG sebanyak 1000 g/mol, manakala luas permukaan adalah 585 m^2 g^{-1} pada suhu pengkarbonan 660 ^oC dan kepekatan PEG berisipadu 31% dengan berat molekul PEG sebanyak 1000 g mol^{-1}. Untuk memastikan semua keputusan ini, sintesis monolit bersalut karbon dijalankan. Hasil yang diperolehi ialah isipadu liang sebanyak 161 mm^3 g^{-1} dan luas permukaan sebanyak 553 m^2 g^{-1}, di mana sangat hampir dengan jangkaan menggunaan RSM.

Prestasi monolit karbon teraktif dinilai menggunakan penjerapan *methylene blue* (MB). Data penjerapan keseimbangan diramalkan melalui tiga garis isoterma,

iaitu isoterma Langmuir, isoterma Freundlich, dan isoterma Redlich-Peterson. Data yang paling sesuai diperolehi melalui isoterma Redlich-Peterson dan isoterma Langmuir dengan pekali-pekali kolerasi (R^2) sebanyak 0.997 dan 0.998. Kapasiti penjerapan maksimum adalah 191 mg/g. Isoterma Langmuir digunakan untuk pemodelan dan simulasi kerana ia adalah model yang mempunyai dua parameter dan mempunyai kejituan yang hampir sama untuk penjelasan data isoterma dalam kajian ini. Parameter keseimbangan tak berdimensi (R_L) dikira sebanyak 0.1, menunjukkan bahawa penjerapan adalah memuaskan.

Kinetik penjerapan MB diselidik dari segi pseudo tertib pertama dan mekanisme tertib kedua untuk penjerapan kimia sepertimana mekanisme resapan intra-partikel dengan menggunakan penghampiran daya pacu linear (LDF) dalam sistem kelompok. Parameter-parameter kinetik dan pekali-pekali kolerasi ditentukan. Dapat ditunjukkan bahawa persamaan kinetik tertib pertama sangat sesuai untuk menjelaskan kinetik-kinetik dengan kadar malar masing-masing 6.6×10^{-3} min^{-1}, 4.0×10^{-3} min^{-1}, dan 1.5×10^{-3} min^{-1} bagi kepekatan awal 20, 50, and 100 mg L^{-1}.

Model LDF untuk sistem monolit dihasilkan. Kadar malar pseudo tertib pertama digunakan sebagai tekaan awal untuk menganggar pekali pemindahan jisim LDF (k_{LDF}) dengan memadankan simulasi dengan data eksperimental. Perbandingan keputusan dikira menggunakan model LDF dan data eksperimental berada dalam persetujuan yang sesuai iaitu k_{LDF} bernilai masing-masing 8.25×10^{-3}, 5.20×10^{-3}, dan 2.50×10^{-3} min^{-1} bagi kepekatan-kepekatan awal 20, 50, dan 100 mg L^{-1}.

Model jangkaaan terserak aliran palam, dengan kadar penjerapan yang dijelaskan oleh model LDF, dibangunkan untuk meramal lengkung bulus turus monolit. Nilai parameter-parameter model diperolehi daripada bahagian eksperimen-eksperimen penjerapan kelompok. Keputusan simulasi didapati sangat bersesuaian dengan data eksperimental. Kesan pekali-pekali pemindahan jisim LDF dikaji secara berangka. Pekali-pekali pemindahan jisim LDF didapati sangat mempengaruhi bentuk lengkung bulus.

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I certify that an Examination Committee has met on 23 October 2009 to conduct the final examination of Darmadi on his Doctor of Philosophy thesis entitled "Preparation of Furfuryl Alcohol Derived Activated Carbon Monolith and Its Adsorption Characteristics of a Cationic Dye" 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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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

DARMADI

Date: 17 June 2009

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LIST OF ABBREVIATIONS/NOTATIONS/SYMBOLS

a	characteristic length of monolithic channel in equation (4.23)	mm
a_L	Langmuir constants (adsorption intensity)	$\mathrm{L\ mg^{-1}}$
$A_{j,m}$	first derivative of the Lagrange interpolation polynomial	
$B_{j,m}$	second derivative of the Lagrange interpolation polynomial	
b_j	Redlich-Peterson isotherm constant	$\mathrm{(L\ mg^{-1})^{\beta}}$
C	Concentration of the solution at time t	$\mathrm{mg\ L^{-1}}$
C_0	Concentration of solute in the solution at equilibrium	$\mathrm{mg\ L^{-1}}$
C_F	feed concentration in monolithic column	$\mathrm{mg\ L^{-1}}$
C^*	equilibrium concentration in liquid phase	$\mathrm{mg\ L^{-1}}$
D_{eff}	effective diffusion coefficient	$\mathrm{cm^2\ s^{-1}}$
D_{ax}	axial dispersion coefficient	$\mathrm{cm^2\ s^{-1}}$
i	an index	
j	an index	
J	order of polynomial in the method of OC	
k	number of factors in equation (3.3)	
k_1	rate constant of pseudo first-order sorption	$\mathrm{min^{-1}}$
k_2	rate constant of pseudo second-order sorption	$\mathrm{g\ (mg\ min)^{-1}}$
K	linear adsorption constant in equation (4.43)	
K_F	Freundlich constant	$\mathrm{mg\ g^{-1} \times (mg\ L^{-1})^{-1/n}}$
k_{LDF}	linear driving force mass transfer coefficient	$\mathrm{min^{-1}}$
K_L	Langmuir constant (adsorption capacity)	$\mathrm{L\ g^{-1}}$
K_j	Redlich-Peterson constant (adsorption capacity)	$\mathrm{L\ g^{-1}}$