UNIVERSITI PUTRA MALAYSIA

ADSORPTION OF CARBON DIOXIDE ON AMMONIA-TREATED CARBON COATED MONOLITH

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By

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ADSORPTION OF CARBON DIOXIDE ON AMMONIA-TREATED CARBON-COATED MONOLITH

By

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JANUARY 2013

Chairman : Professor Thomas Choong Shean Yaw, PhD, IR

Faculty : Engineering

Due to the greenhouse effect, the reduction and recovery technology of carbon dioxide has become an important research topic nowadays. In addition to the researches on the use of carbon dioxide as a reactant, the recovery and concentration of carbon dioxide from waste gas are the key issues. The adsorption method that is used to capture CO_2, is in a packed-bed column.

In this study, a monolithic column designed to separate and concentrate CO_2 from He/CO_2 mixture, was used to study the feasibility for recovery of CO_2 from gaseous mixture. The surface properties of carbon-coated monolith were modified by N-containing group of ammonia aqueous and potassium hydroxide to increase CO_2 adsorption and then compare it with untreated Carbon-Coated Monolith's (CCM) capacity. BET, EDX, SEM, FT-IR, and Boehm titration methods were used to identify the physical and chemical properties of modified and un-treated CCM.

The adsorption capacity and characteristics of carbon dioxide on carbon-coated monolith have been measured out in a variety of temperatures (303, 313, and 323 K), pressure (100,150, and 200 kPa), flow rate (15 to 480 mL/min), and variety of concentration (5 to 35%) of CO_2 in feed.

Modification by ammonia aqueous and potassium hydroxide as an N-containing method were treated to enhance the basicity of carbon surface. Different concentrations of ammonia were applied to introduce some functional groups such as (N-H) on surface so that it would increase adsorption capacity. Concentration of CO_2 output from the carbon-coated monolith packed column is a function of time that was indicated by an analyzer.

The adsorption capacity showed increase adsorption with increasing input flow rate, pressure, and concentration, and reduction of CO_2 adsorption capacity with rising temperature up to 323 K. Modified CCM (ACCM-75) showed an increased adsorption capacity of CO_2 by around 12 % and PCCM showed an increase of almost 27 % compared to untreated CCM.

The Deactivation Model (DM) derived using the analogy between the adsorption of CO_2 and the deactivation of catalyst particles. Observed adsorption rate constants (K_s) and first-order deactivation rate constant (K_d) were obtained from the model. It was found that the DM describes experimental breakthrough curves very well. The isotherm was fitted well with the Toth and Langmuir equations.

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

PENJERAPAN KARBON DIOKSIDA KE ATAS AMMONIA-DIRAWAT CARBON-BERSALUT MONOLIT

Oleh

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JANUARY 2013

Pengerusi : **Profesor Thomas Choong Shean Yaw, PhD, IR**

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Disebabkan oleh kesan rumah hijau, teknologi pengurangan dan pemulihan karbon dioksida menjadi topik penyelidikan yang penting pada masa kini. Di samping kajian tentang menggunakan karbon dioksida sebagai reaktan, pemulihan dan kepekatan karbon dioksida daripada gas sisa adalah isu utama. Kaedah penjerapan yang digunakan untuk menangkap CO_2, adalah dalam ruang-katil tetap.

Dalam kajian ini, kolum monolitik direka untuk memisahkan dan menumpukan CO_2 daripada He/CO_2 campuran, telah digunakan untuk mengkaji kemungkinan CO_2 pemulihan daripada campuran gas. Ini permukaan hartanah meningkatkan monolit bersalut karbon oleh diubah suai kumpulan N-mengandungi ammonia dan kalium hidroksida untuk meningkatkan CO_2 penjerapan dan kemudian dibandingkan dengan kapasiti Karbon kapasiti Monolith (CCM) tidak dirawat.

BET, EDX, SEM, FT-IR, dan Boehm titratan telah digunakan untuk mengenal pasti ciri-ciri fizikal dan kimia diubahsuai CCM dan tidak dirawat.

Kapasiti penyerapan dan ciri-ciri karbon dioksida pada bersalut karbon monolit telah diukur dalam suhu kepekatan (303, 313, dan 323 K), tekanan (100,150, dan 200 kPa), kadar aliran (15-480 mL / min), dan kepekatan tumpuan (5 hingga 35%) CO_2 dalam suapan.

Pengubahsuaian oleh akueus ammonia dan kalium hidroksida sebagai satu kaedah yang mengandungi N-telah dirawat untuk meningkatkan kebesan permukaan karbon. Perbezaan kepekatan ammonia telah digunakan untuk memperkenalkan beberapa kumpulan berfungsi seperti (NH) pada permukaan bahawa ia akan meningkatkan kapasiti penjerapan. Kepekatan pengeluaran CO_2 dari ruang bersalut karbon monolit dibungkus adalah satu fungsi masa seperti yang telah ditunjukkan oleh penganalisis.

Kapasiti penjerapan menunjukkan meningkatkan penjerapan dengan input meningkatkan aliran kadar, tekanan, dan penumpuan, dan mengurangkan CO_2 kapasiti penjerapan dengan peningkatan suhu sehingga kepada 323 K. Dirawat CCM (ACCM-75) menunjukkan peningkatan kapasiti penyerapan CO_2 oleh sekitar 12% dan PCCM menunjukkan peningkatan hampir 27% berbanding kepada CCM tidak dirawat.

Model Penyahaktifan (DM) diperolehi menggunakan analogi antara penjerapan CO_2 dan penyahaktifan zarah pemangkin. Perfileman pemalar kadar penjerapan (*Ks*) dan kadar tertib pertama penyahaktifan berterusan (*Kd*) telah diperolehi daripada model. Ia telah mendapati bahawa DM menerangkan keluk kejayaan eksperimen sangat baik. Isoterma telah dipasang dengan baik dengan Toth dan persamaan Langmuir.

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At long last, I would like to dedicate this thesis to my parents and my love (O_2). You will always be the source of my inspiration and a part of me.

I certify that a Thesis Examination Committee has met on 18 January 2013 to conduct the final examination of Ehsan Marahel on his thesis entitled “Adsorption of Carbon Dioxide by Activated Carbon Modified with the Mixture of Cu/Zn" in accordance with the Universities and University College 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 Master of Science.

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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 or concurrently submitted for any other degree at Universiti Putra Malaysia or other institutions.

EHSAN MARAHEL

Date: 18 January 2013

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LIST OF ABBREVIATIONS/NOTATIONS/SYMBOLS

Nomenclatures

Symbols		Unit
A_{cs}	Projected area covered by a nitrogen molecule (0.162 nm^2)	nm^2
b	Toth Parameter	kPa
C	CO_2 exit concentration at any time	$kmol.m^{-3}$
C_o	CO_2 feedstock concentration	$kmol.m^{-3}$
F	Total flow rate	$mL.min^{-1}$
k	Langmuir isotherm constant	L/g
K_d	First-order deactivation rate constant	S^{-1}
K_g	Mass-transfer coefficient of the gas film	ms^{-1}
K_s	observed adsorption rate constant	ms^{-1}
N	Avogadro number (6.02×10^{23})	mol^{-1}
P	Pressure	kPa
q_{max}	Toth's maximum adsorption capacity	$mmol.g^{-1}$

q	Adsorption Capacity	mol.kg^{-1}
Q_o	volumetric flow rate of CO_2-rich gas	m^3 s^{-1}
S	vacant surface area of the adsorbent at any time	m^2
S_{BET}	BET surface area	m^{2}g^{-1}
T	Temperature	^{o}C or K
t	Time	s
t_b	Breakthrough time	s
τ	surface-time (S_o/Q_o)	s.m^{-1}
W	Weight of adsorbent	g

Abbreviations

ACCM	Ammonia Carbon-Coated Monolith
BET	Brunauer-Emmett-Teller
BJH	Barret-Joyner-Halenda
CCM	Carbon-Coated Monolith
CFC	Chlorofluorocarbon
Cpsi	Cells per square inch
CVD	Chemical Vapor Deposition
DR	Dubinin-Radushkevich
DM	Deactivation Model
DFT	Density Functional Theories
EDX	Energy-dispersive X-ray
FA	Furfuryl Alcohol
FTIR	Fourier Transform Infrared Spectroscopy
GHG	Green House Gas
IPCC	Intergovernmental Panel on Climate Change
IUPAC	International Union of Pure and Applied Chemistry
MTZ	Mass Transfer Zone
PEG	Poly Ethylene Glycol
PSA	Pressure Swing Adsorption
PCCM	Potassium hydroxide Carbon-Coated Monolith
Ppm	Parts per million
SBA	Santa Barbar-University of California
SEM	Scanning Electron Microscopy
TSA	Temperature Swing Adsorption

CHAPTER 1

INTRODUCTION

1.1 Motivation

Since the industrial revolution, the use of fossil fuels has considerably increased over the years. Nowadays, fossil fuels supply more than 98 % of the world's demand (Siriwardane et al., 2001; Socolow et al., 2004). The rise in global temperature is widely attributed to an increase in greenhouse gases (GHG) in the atmosphere. The most abundant greenhouse gas –CO_2– has risen from preindustrial levels of 280 parts per million (ppm) to present levels of over 385 ppm. Main emissions of CO_2 come from the combustion of fossil fuels such as coal, natural gas or petroleum, and industrial processes such as oil refinement, the production of cement, iron, and steel as shown in Figure 1.1 (Pireset al., 2001; Behvandi and Tourani, 2011).

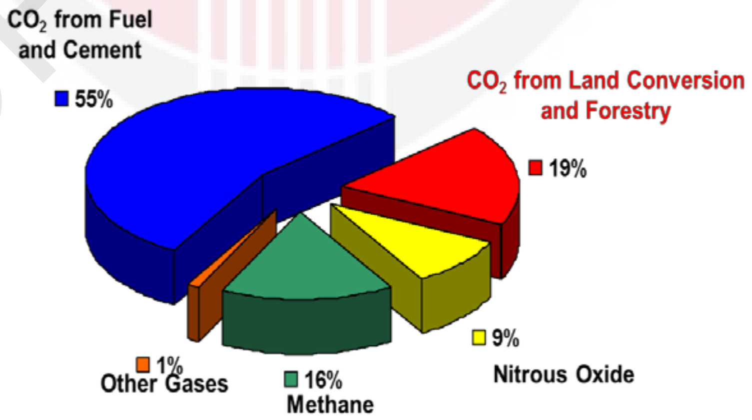

Figure 1.1: Emissions of CO_2 (Jung and Shiang, 2005)

Improving the energy efficiency of fossil-fired power generation is one of the fundamental measures needed to reduce greenhouse gases emissions. Other techniques to reduce CO_2 emissions into the atmosphere include: (i) the replacement of fossil fuels with cleaner fuels or fuels with lower carbon content or nuclear energy, (ii) increasing the use of fuels from renewable energy sources such as biomass, solar and wind energy, and (iii) capturing and storing CO_2. The high costs of renewable energies associated with the abundance and availability of fossil fuels delay the introduction of these environmental friendly energy forms. All these methods include the attractive feature of limiting the amount of carbon dioxide emitted into the atmosphere, but not without economic, technical, or societal limitations (Burchell and Judkins, 1996; Lund, 2006; Allwar et al., 2008).

According to Herzog and Golomb (2004), from the economical point of view, capture and storage is an appropriate approach. A number of techniques could be used for the separation of carbon dioxide from fuel gas streams. The chemical absorption process can control carbon dioxide with higher removal efficiency, but it is a very energy intensive process and is accompanied with the problem with corrosion. Other techniques considered, are cryogenic separation, membrane separation, and adsorption processes, including pressure swing adsorption, vacuum swing adsorption, temperature swing adsorption and electric swing adsorption. The CO_2 capture by adsorption is an attractive technique as a post-combustion treatment of flue gas (Gomes and Yee, 2002).

Adsorption processes usually appear in the context of high selectivity towards CO_2 with the tendency of reducing energy penalties and is a simple and inexpensive operation for removing process gases and vapors from the air, by passing them through an adsorption bed packed with a porous adsorbent (Arenillas et al., 2005; Bonenfant et al., 2008; Safaai et al., 2010).

Porous silica, alumina, zeolite, and activated carbon are used as adsorbents for a wide range of applications such as CO_2 adsorption. The ideal sorbent would present high adsorption capacity and selectivity for carbon dioxide, and be easily regenerated in order to be economically feasible. The affinity of the adsorbent for carbon dioxide should not be too high; otherwise, the regeneration step will negatively affect the economy of the process (Drage et al., 2009).

In traditional adsorption processes, packed beds are used due to the inexpensive and adaptable adsorbent. However, it created high mass transfer resistance and a pressure drop at high flow rate. In air pollution control processes, large volumes of gases have to be treated, which require the reactor to have small pressure drops, in these cases, the use of activated carbon monoliths is very favorable (Chen et al., 2002; Chang et al., 2003). Activated carbon monolith has been fabricated by coating carbonaceous materials onto cordierite monolith or with full-body extrusion. The monolith shaped structured adsorbent carbon monoliths, are characterized by straight parallel channels separated by thin walls, high void fraction and large geometric surface area, resulting in a low pressure drop under high flow rate and large contact area. These properties make carbon monoliths attractive (Ruthven and Thaeron, 1996; Menard et al., 2005; Thiruvenkatachari et al., 2009).

The monolith has an open structure, which, allows for higher flow of fluids through them with a lower drop in pressure so that CO_2 can occupy the micropores where they may be selectively adsorbed. These monoliths present the advantages of superior contact efficiency and a higher adsorption capacity with respect to granular activated carbons (Liu et al., 2007; Boonpoke et al., 2011).

The basicity of the carbon comes from basic groups or basic nitrogen containing groups on the surface of carbon. They would produce more adsorption sites for CO_2 adsorption if there were more basic nitrogen-containing groups on the surfaces. Thus, it was inferred that the surface of basic chemical groups on the carbon-coated monolith sample played a key role in the enhancement of CO_2 adsorption. So, in order to improve the adsorption performance of the Carbon-Coated Monolith (CCM) for CO_2, the application of surface modification is used effectively to increase the surface of basic groups and decrease the acidic oxygen-containing groups' surfaces (Chen et al., 2005).

1.2 Problem Statement

In the adsorption application, activated carbon is generally used in powder and granular forms. The use of carbon in these forms for column is associated with high-pressure drops, potential channeling, and other disadvantages. The problem encountered can be overcome by the use of carbon monoliths. Compared to the conventional fixed bed column, monolithic columns provide the advantages of low-pressure drop, larger external surface area, and short diffusion lengths (Edwards and Riggs, 2003; Moreno-Castilla, 2010).

It can also be located in a vertical and horizontal position or in mobile system without losing shape and is easier to be scaled up due to its simple design and uniform flow-distribution

(Allwar et al., 2008; Khalil et al., 2011). The surface of carbon can be improved by so many chemicals and methods. N-containing is an effective method, which can increase the amount of CO_2 adsorption (Figueiredo et al., 1999; Przepiorski, 2006; Shafeeyan et al., 2011).

1.3 Objective

The main objective of this project is to develop a carbon-coated monolith using Furfuryl Alcohol (FA) as carbon source, PEG as pore former, pyrrole as binder and HNO_3 as catalyst and the utilization of carbon-coated monolith in CO_2 adsorption system. A monolithic column system is used to study the amount of CO_2 removal. Both experimental and modeling work is carried out. Several objectives are listed below:

a) To prepare carbon-coated monolith by using furfuryl alcohol via dip-coating method.

b) To investigate the efficiency of carbon dioxide adsorption by carbon-coated monolith samples and, modification with NH_3 and KOH.

1.4 Scope of work

The main scopes in this investigation, is to achieve the specified objectives comprises of research activities such as preparation of Carbon-coated Monolith and modification, the characterization of the adsorbents and the adsorption capacity study in different operation conditions for carbon dioxide on the carbon-coated monolith.

Carbon-coated monolith are modified with different amount of ammonia aqueous (from 25 to 100 mL per gram carbon), to see the effect of modification on the breakthrough curve and the adsorption capacity.

The carbon-coated monolith and its modified were characterized by different techniques. Boehm's titration was used to calculate the concentration of functional groups on carbon-coated monolith. Scanning electron microscopy (SEM) was used to determine surface morphology of the carbon-coated monolith and treatment with NH_3 and KOH. The characterization of chemical structure of carbon samples were performed using FT-IR spectroscopy method. The specific surface area and pore volume of activated carbon samples were estimated by BET analysis. In addition, the amounts of new elements on samples from preparation and modification process were characterized by EDX analysis.

Other than adsorption capacity study on carbon-coated monolith, the operation condition that have the best adsorption capacity is used as the standard condition to investigate of series of NH_3 and KOH modification on the carbon-coated monolith.

1.5 Outline

The present work has been categorized into five chapters. A concise note of the hazardous effects of greenhouse gases (CO_2), the aims of the study and complete schematics of the research is contained in chapter I. A literature review of CO_2 adsorption in a packed-bed of different adsorbents, theoretical method of breakthrough, isotherm models and characterization methods are presented in chapter II.

The experiments as well as theoretical techniques used and characterization methods have been studied thoroughly in chapter III. While chapter IV, addresses the adsorption of CO_2 on untreated and treated carbon-coated monolith. The results regarding the characterization of samples, breakthrough curves at different conditions and comparison between the equilibrium data with theoretical isotherm models were also discussed. Moreover, finally, conclusions and recommendations for further work are summarized in chapter V.

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