

**LIQUID PHASE ADSORPTION ISOTHERM OF
AZEOTROPIC AND NON-AZEOTROPIC ORGANIC
SOLVENT MIXTURE ONTO POROUS ADSORBENTS**



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**FACULTY OF ENGINEERING
UNIVERSITI MALAYSIA SABAH
2015**

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**THIS IS SUBMITTED IN PARTIAL FULFILLMENT
FOR THE DEGREE OF MASTER OF ENGINEERING**

**FACULTY OF ENGINEERING
UNIVERSITI MALAYSIA SABAH
2015**

UNIVERSITI MALAYSIA SABAH

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ONTO POROUS ADSORBENTS**

DEGREE : **MASTER OF ENGINEERING (CHEMICAL ENGINEERING)**

VIVA DATE : **27th AUGUST 2014**



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ACKNOWLEDGEMENT

First and foremost, I would like to thank Allah SWT who Has blessed me and guided me until I am able to accomplish this thesis as a partial fulfilment of the requirements for the Master Degree of Chemical Engineering in Universiti Malaysia Sabah (UMS).

I am heartily thankful to my supervisor, Prof Dr. Awang Bono and my co-supervisor, Mdm. Farm Yan Yan, whose encouragement, guidance, support, valuable time, advice, criticism and correction to this thesis from the beginning up to the end of the writing. I also want to thank all lecturers of the Faculty of Engineering in UMS who have taught and guided me during the years of study in this faculty.

In this very special moment, I would like to express my deepest thanks to my research partner, Norina Yadin, for the moral support and having confidence in me throughout the research work. Moreover, to the lab assistants, Ms. Noor Aemi Dawalih, Mdm. Noridah Abas and Mr. Razis, for assisting me in the laboratory until I managed to complete my lab works successfully.

Furthermore, to my beloved parents and family, for their love encouragement and supports both financially and mentally that made me possible to finish my study.

Lastly, I offer my regards and blessings to all of those who supported me in any respect during the completion of this research.

ABSTRACT

Adsorption isotherm is an essential component in the understanding of the adsorption process. There are several ways and methods for the measurements, analysis and interpretation of adsorption isotherms either for gas or liquid phase adsorption. However, in the adsorption from solution were usually conducted at low region of liquid concentration and the direct interpretation of excess adsorption isotherm as adsorption isotherm. Therefore, a study on the adsorption of azeotropic and non-azeotropic organic solvent mixture onto porous adsorbent for whole range of liquid concentration is conducted. The study includes the measurement of excess adsorption isotherm, theoretical analysis, and interpretation of adsorption isotherm by using Pseudo Ideal Adsorption Model, Gibbs Dividing Plane Model and Langmuir-Freundlich Model as well as validation of theoretical analysis by direct measurement of adsorption. In this study, organic solvents such as acetone, methanol and propanol were chosen as the adsorbates whereas activated carbon and silicalite were chosen as the porous adsorbents. The measurement of excess isotherm was conducted using conventional method where known amount of adsorbent was mixed with known quantity and quality of organic solvent mixture in a sealed container and kept at constant temperature for a certain period to be equilibrated. Then the equilibrated liquid concentration was determined by standard procedure using Gas Chromatography Mass Spectrometer. The result of this study had shown that some agreement for the complete concentration range of adsorption isotherm trend for those interpreted and measured values, but not the magnitude.

ABSTRAK

FASA CECAIR PENJERAPAN ISOTERMA UNTUK CAMPURAN LARUTAN ORGANIK AZEOTROP DAN BUKAN AZEOTROP KE ATAS PENJERAP BERLIANG

Penjerapan isoterma merupakan komponen penting dalam memahami proses penjerapan . Terdapat beberapa cara dan kaedah dalam pengukuran, analisis dan tafsiran penjerapan isoterma sama ada untuk penjerapan gas atau cecair. Walau bagaimanapun, penjerapan cecair biasanya dijalankan pada julat kepekatan yang rendah dan tafsiran langsung daripada lebih penjerapan isoterma sebagai penjerapan isoterma sering dilakukan. Oleh itu, kajian mengenai penjerapan campuran pelarut organik azeotrop dan bukan azeotrop ke atas dua penjerap yang berbeza struktur liang untuk keseluruhan julat kepekatan cecair telah dijalankan. Kajian ini termasuk pengukuran lebih penjerapan isoterma , analisis teori, dan tafsiran isoterma penjerapan berdasarkan teori Pseudo Ideal Adsorption Model, Gibbs Dividing Plane Model dan Langmuir-Freundlich Model serta pengesahan analisis teori dengan ukuran langsung penjerapan . Dalam kajian ini, pelarut organik seperti aseton, metanol dan propanol telah dipilih sebagai komponen yang dijerap manakala karbon diaktifkan dan silikalit telah dipilih sebagai penjerap. Pengukuran lebih isoterma dijalankan dengan menggunakan kaedah konvensional di mana sejumlah kuantiti penjerap dicampurkan dengan campuran pelarut organik di dalam bekas bertutup dan disimpan pada suhu yang berterusan untuk tempoh masa yang tertentu untuk mencapai keseimbangan. Kemudian kepekatan cecair dianalisis menggunakan Gas Chromatography Mass Spectrometer. Hasil kajian ini telah menunjukkan bahawa lebih penjerapan isoterma yang ditafsirkan terus dari model penjerapan dan nilai-nilai yang diukur secara konvensional untuk julat kepekatan yang lengkap mempunyai trend yang sama tetapi berbeza dari segi magnitud.

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LIST OF ABBREVIATIONS

ZSM-5	-	Zeolite Socony Mobil-5
TCD	-	Thermal Conductivity Detector
psi	-	Pounds per square inch
GCMS	-	Gas Chromatography Mass Spectrophotometer

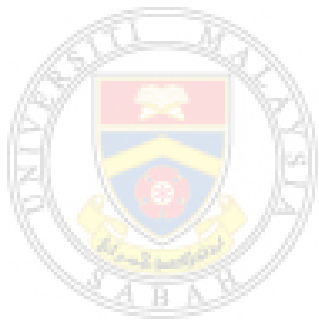


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LIST OF SYMBOLS

C_0	-	<i>initial concentration of adsorbate in mg/L</i>
C_e	-	<i>final equilibrium concentration of adsorbate in mg/L</i>
m	-	<i>mass of adsorbent in g</i>
N_s	-	<i>saturation adsorption capacity in mole/g</i>
W_0^w	-	<i>initial total mass of liquid mixture in g</i>
W_s	-	<i>mass of adsorbent in g</i>
W_0	-	<i>initial total mole of liquid mixture in mole</i>
V	-	<i>volume of liquid mixture in L</i>
Γ_1^w	-	excess adsorption of component 1 in m/g
Γ_1	-	excess adsorption of component 1 in mole/g
$\Gamma(C_e)$	-	excess adsorption value
a_{\max}	-	maximum adsorbate amount which could be adsorbed on 1 m ² on the adsorbent surface
a_{tot}	-	total adsorbed amount
c	-	BET constant
$C_e \cdot V_a$	-	equilibrium adsorbate concentration on the volume of adsorbed layer
E_1	-	heat of adsorption for the first layer
E_L	-	that for the second and higher layers and is equal to the heat of liquefaction
K	-	equilibrium constant
K_0	-	the preexponential factor
n_1^s	-	amount of component 1 adsorbed
n_2^s	-	amount of component 2 adsorbed
p	-	equilibrium pressure of adsorbates at the temperature of adsorption
p_0	-	saturation pressure of adsorbates at the temperature of adsorption
q	-	adsorbent phase concentration after equilibrium
x_{10}^w	-	initial mass fraction
x_1^w	-	equilibrium mass fraction
x_{10}	-	initial liquid mole fraction of component 1
x_1	-	equilibrium liquid mole fraction of component 1
x_2	-	equilibrium liquid mole fraction of component 2

- x_1^s - adsorbed phase mole fraction of component 1
- v - the adsorbed gas quantity
- v_m - the monolayer adsorbed gas quantity
- V_a - specific adsorbed volume of sorbates



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CHAPTER 1

INTRODUCTION

1.1 Background

Adsorption is the phenomenon of accumulation of large number of molecular species at the surface of liquid or solid phase in comparison to the bulk. The process of adsorption arises due to presence of unbalanced or residual forces at the surface of liquid or solid phase. These unbalanced residual forces have tendency to attract and retain the molecular species with which it comes in contact with the surface. Adsorption is essentially a surface phenomenon. Adsorption is a term which is completely different from absorption. Meanwhile, absorption means uniform distribution of the substance throughout the bulk, adsorption essentially happens at the surface of the substance.

There are many important applications of adsorption process in industries such as color removal in textile industries. Hameed and co-workers (Hameed *et al.*, 2007) applied oil-palm ash as adsorbent in eliminating dyes from textile wastewater since Malaysia is the largest exporter of palm oil in the international market make it a low-cost and easily available adsorbent. Other than that, it also helps in removing color and taste as stated in the literature (Hanzlík *et al.*, 2004; Juang *et al.*, 2006). Other application includes the removal the unwanted organic and inorganic impurities in drinking waters and waste effluence (Juang *et al.*, 2006; Abdelwahab, 2008).

The potential theory of adsorption first introduced in 1914 by Polanyi (Bansal & Goyal, 2005) and it has now been widely employed to remove unwanted materials from a solution (Hameed *et al.*, 2007; Rahchamani *et al.*, 2011; Ahmed & Theydan, 2012). The adsorption process is divided into physical adsorption (Saha *et al.*, 2003; Critoph & Metcalf, 2004) and chemical adsorption (Srivastava & Eames, 1998; Kato *et al.*, 2001).

In the case of physical adsorption or physisorption, the adsorbate is bound to the surface by relatively weak van der Waals forces, which are similar to the molecular forces of cohesion and are involved in the condensation of vapors into liquids (Bansal & Goyal, 2005). The performance of adsorbents used in physisorption is governed largely by surface properties, such as surface area, micropores and macropores, size of granules in powders, crystals or in pellets (Srivastava & Eames, 1998). Physical adsorbents with mesopores can adsorb consecutive layers of adsorbate, while those with micropores, have the volume of the pores filled with the adsorbate (Wang *et al.*, 2009). Chemisorption or chemical adsorption, on the other hand, involves exchange or sharing of electrons between the adsorbate molecules and the surface of the adsorbent resulting in a chemical reaction (Bansal & Goyal, 2005). Chemical adsorption is caused by the reaction between adsorbates and the surface molecules of adsorbents. Electron transfer, atom rearrangement and fracture or formation of chemical bond always occurs in the process of chemical adsorption. Only one layer of adsorbate reacts with the surface molecules of chemical adsorbent (Wang *et al.*, 2009).

Adsorbent plays the major role in influencing the adsorption process ability. Most of the solid adsorbents of great industrial applications possess a complex porous structure that consists of pores of different sizes and shapes. In terms of the experience of adsorption science, total porosity is usually classified into three groups. According to the IUPAC recommendation, the micropores are defined as pores of a width not exceeding 2 nm, mesopores are pores of a width between 2 and 50 nm, but macropores represent pores of a width greater than 50 nm. The significance of pores in the adsorption processes largely depends on their sizes. Since sizes of micropores are comparable to those of adsorbate molecules, all atoms or molecules of the adsorbent can interact with the adsorbate species. That is the fundamental difference between adsorption in micropores and larger pores like meso- and macropores (Dabrowski, 2001). Thus in this research work, activated carbon and silicalite were selected as the adsorbents. The selection of the adsorbent was based on pore structure.