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COMPARING THE REMOVAL PERFORMANCE OF CO₂ GAS USING ADSORPTION ABSORPTION TECHNIQUES

NURULHAIDA BINTI LUHID

A Final Year Project Report submitted in partial fulfillment of the requirements for the award of the degree of Bachelor of Chemical Engineering (Gas Technology)

Faculty of Chemical Engineering & Natural Resources Universiti Malaysia Pahang

APRIL 2008

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To my family

Thank you for the support, encouragement and motivation that have been given

To all my friends

Thank you for the support and assistance that have been given

ACKNOWLEDGEMENT

In the early stage of my research and in organizing my thoughts on the thesis, I have benefited greatly from discussions with a number of researchers, academicians and practitioners. In particular, I wish to express my sincere appreciation to my main thesis supervisor, Dr. Hayder A. Abdul Bari for encouragement, guidance, critics and friendship.

Beside that, I would like to express my thanks to Mr. Khairil Anuar Abdul Hamid and Mr. Mohd Anuar Hj Ramli as the Teaching Engineer in Unit Operation Lab for their guidance and technical support. Without their continued support and interest, this thesis would not have been the same as presented here.

My fellow friends especially Ng Wei Kuen and Qusyairi Ali should also be recognized for their support. Their views and support are useful indeed. Unfortunately, it is not possible to list all of them in this limited space.

Last but not least, my loving mother and father respectively, Madam Tengku Maimunah Tengku Omar and Mr. Luhid Daud who are very supportive morally. Not to forget the lecturers from Faculty of Chemical and Natural Resources Engineering who had teach me all this while.

ABSTRACT

Over the past century, the Earth has increased in temperature by about 5 °C and this is because of an increase in concentration of the main greenhouse gases; carbon dioxide. Therefore, a modification of gas phase of adsorption-absorption process was used. In the recent research, this technique was established by using a single adsorbent. To compare the effect of changing the gas removal techniques and to study the performance on the CO₂ gas removal from different adsorbent, Gas Absorption Adsorption Unit was used with granular activated carbon and zeolites as the adsorbents. For this purpose, purified CO₂ was used. This study was conducted using various gas flow rate (100, 75 and 50 m³/hr) and water flow rate (250 and 200 L/hr). The experiment had been run twice at different condition; with present and absent of zeolites. At the end of the study, it was observed that increasing of gas flow rate reduce the composition of CO₂ dissolved in the water. On the other hand, increasing the water flow rate will increase the composition of CO₂ dissolved in the water solution. Beside that, higher gas flow rate increases the efficiency of the process. Increasing of water flow rate will increase the CO₂ composition dissolved even greater rather than increasing gas flow rate. The CO₂ composition dissolved in water is decrease with the present of zeolites. This shows that by having zeolites, it helps to decrease the rate of absorption of CO2. The process will be more efficient if the CO₂ composition in the receiving vessel is high.

ABSTRAK

Sejak beberapa abad yang lalu, suhu Bumi telah meningkat sebanyak 5°C disebabkan oleh kandungan Gas Rumah Hijau yang tinggi; karbon dioksida. Untuk itu, pembaharuan dalam proses penyerapan-penjeraban gas dilakukan. Jika dibandingkan dengan kajian-kajian yang lalu, teknik menggunakan hanya satu jenis penyerap banyak dijalankan. Bagi membandingkan kesan perubahan teknik gas dan mempelajari perbezaan pencapaian gas pada penyerap yang berbeza, 'Gas Absorption Adsorption Unit' telah digunakan bersama-sama dengan karbon aktif granul dan zeolite sebagai penyerap. Untuk itu, gas karbon dioksida tulen telah digunakan. Kajian telah dijalankan menggunakan variasi kadar alir gas (100, 75 and 50 m³/j) and kadar alir air (250 and 200 L/j). Ujikaji telah dijalankan sebanyak dua kali pada keadaaan yang berbeza; dengan kehadiran dan tanpa zeolite. Pada akhir kajian, didapati bahawa peningkatan kadar alir gas mengurangkan komposisi gas karbon dioksida yang larut dalam air. Dalam erti kata yang lain, peningkatan kadar alir air menyebabkan peningkatan komposisi karbon dioksida yang larut dalam air. Selain itu, kadar alir gas yang tinggi menyebabkan keberkesanan proses meningkat. Apabila kadar alir air meningkat, komposisi karbon dioksida yang larut turut meningkat malah lebih besar dari peningkatan kadar alir gas. Komposisi karbon dioksida larut dalam air menurun dengan kehadiran zeolite. Ini menunjukkan dengan adanya zeolite, membantu kepada penurunan kadar penjeraban karbon dioksida. Kajian ini akan lebih efisyen sekiranya komposisi karbon dioksida pada vesel permulaan berada pada kedudukan yang tinggi.

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LIST OF SYMBOLS

Р	-	Pressure
P/P _o	-	Partial pressure
W	-	Width
V3	-	Valve
B1	-	Feed vessel
K1	-	Packed column
P1	-	Pump

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The 20th century has seen rapid increase of population and explosive growth in energy consumption. As more countries becoming industrialized, it is expected that more energy will be consumed in 21^{st} century. EIA predicts 57 percent increase of energy demand from 2004 to 2030 (EIA, 2007). Table 1.1 shows the comparisons of energy use, population and per capita consumption in 1900 and 2001 (Song C, 2006). In current stage over 85 percent of world energy demand is supplied by fossil fuels. Fossil-fueled power plants are responsible for roughly 40 percent of total CO₂ emissions, coal-fired plants being the main contributor (Carapellucci and Milazzo, 2003).

Environmental issues due to emissions of pollutants from combustion of fossil fuels have become global problems, including air toxics and greenhouse gases (GHG). The CO_2 emission from human activity was on the order of 7 Gt/a in the late 1990's (Yamasaki, 2003). This includes the combustion of fossil fuels in all major industries and other factors such as deforestation and desertification.

Energy Source	1900) Use	2001 Use	
	MTOE ^a	% or Unit	MTOE ^a	% or Unit
Coal	501	55	2395	24
Petroleum	18	2	3913	39
Natural Gas	9	1	2328	23
Nuclear	0	0	662	6
Renewable ^b	383	42	750	8
Total	911	100%	1004.8	100%
Population	1762	Million	6153	Million
Per capita E	0.571	TOE ^a	1633	TOE
use				
Global CO ₂	534	MMTCE ^a	6607	MMTCE
emission				
Per capita CO ₂	0.30	MTCE	1.07	MTCE
emission				
Atmospheric	295	ppmv ^a	371	ppmv
CO ₂				
Life	47.3	Years	77.2	Years
expectancy				
^a TOE: Ton oil e	quivalent; MTOI	E: million ton of	oil equivalent; N	IMTCE: million
metric ton of carbon equivalent; MTCE: metric ton of carbon equivalent; ppmv: part				

Table 1.1: World energy use, population and per capita in 1990 and 2001(Song C, 2006)

^aTOE: Ton oil equivalent; MTOE: million ton of oil equivalent; MMTCE: million metric ton of carbon equivalent; MTCE: metric ton of carbon equivalent; ppmv: part per million by volume; ^b including hydroelectric power, biomass, geothermal, solar and wind energy.

The total amount of carbon on earth is constant and its distribution among lithosphere, atmosphere and biosphere was relatively balanced until the advent of era of industrialized civilization. The CO_2 concentration in the atmosphere is increasing. Figure 1.1 shows the change of atmospheric CO_2 level over the years between 1000 and 1997 and actual CO_2 level during 1958–2004 (Song C, 2006). CO_2 level increased from 280 ppmv in 1000 to 295 ppmv in 1900 based on Antarctica ice core data. It increased to 315 ppmv in 1958 and further to 377 ppmv in 2004 based on actual data logged in Hawaii.



Figure 1.1: Atmospheric CO_2 concentrations during 1000–2004 based on the analysis of ice cores and logged atmospheric CO_2 concentrations during 1958–2004 (Song C, 2006).

Capture of CO_2 contributes 75 percent to the overall CCS cost and CCS increases the electricity production cost by 50 percent (Feron and Hendriks, 2005). Although these numbers may vary with different CCS schemes, cutting the capture cost is the most important issue for the CCS process to be acceptable to the energy industry. Hence, this study mainly focuses on the progress in technologies of CO_2 separation from the chemical conversion point of view. There are many options for CO_2 separation and these include adsorption, absorption, membrane and biotechnology.

1.2 Adsorption

Adsorption is defined as the formation of a layer of gas, liquid, or solid on the surface of a solid or, less frequently of a liquid. There are two types depending on the nature of the forces involved. In chemistry a single layer of molecules, atoms, or ions is attached to the adsorbent surface by chemical bonds. When an adsorbent attracts molecules from the gas, the molecules become concentrated on the surface of

the adsorbent and are removed from the gas phase. In physisorption adsorbed molecules are held by the weaker van der Waals' forces. (Oxford Dictionary of Chemistry, 2000). Adsorptions involve the binding of molecules from their liquid or gaseous environment onto the surface of solids. It is a separation process for the selective removal small quantities of components from a fluid mixture or solution. (Ralph T. Yang, 1997). Adsorption is the process of transferring material from a fluid phase to a solid phase. (W. John Thomas, 1998).

1.3 Absorption

Gas absorption is a process in which soluble components of gas mixture are dissolved in a liquid phase. The gas and liquid normally flow counter currently among some packing which serve to provide the contacting of interfacial surface through which mass transfer take place. (Sunggyu Lee, 2006). Absorption is the take up of a gas by liquid, or the take up of a liquid by a solid. Absorption differs from adsorption in that the absorbed substance permeates the bulk of the absorbing substance; (taken up by volume not by surface). Overall, gas absorption maybe described as the partition of gas between gas and liquid phases. This partition or absorption of a gas is generally discussed in terms of the equilibrium between soluble gas in the gas phase and dissolved gas in the liquid phase is a process of simultaneous mass and heat transfer. (Roman Zarzycki, 1993).

1.4 Problem Statement

The growth in scale of gas-phase adsorption separation processes, particularly since 1970 is due to continuing discoveries of new, porous, high surfacearea adsorbent materials (particularly molecular sieve zeolites) and especially to improvements in the design and modification of adsorbents. These advances have encouraged parallel inventions of new process concepts.

Chemical absorption of CO_2 in a packed column will be very effective using various adsorbent. (D.Georgiou, 1999). Many research have been conducted in recent years on the subject of gas adsorption using a single adsorbent such as activated carbon, zeolite, silica gel, activated alumina and lime soda. But in this study, the removal performance of CO_2 gas is compare by using two adsorbent; granular activated carbon and silver exchanged zeolite.

1.5 Objectives

The objectives of this study will focus on:

- To compare the effect of changing the gas removal techniques using various gas flow rate (100, 75 and 50 m³/hr) and water flow rate (250 and 200 L/hr) at constant pressure 1 bar with temperature 37°C.
- ii. To study the performance on the CO_2 gas removal from different adsorbent; with the present and absent of zeolites.

1.6 Scope of Study

In this study, the removal performances of CO_2 gas will concentrate on adsorption and absorption techniques using Gas Adsorption-Absorption Unit model CE130. The experiment had been run at the constant pressure and temperature (1 bar and 37°C). The performance is analyzed by comparing the liquid flow rate at 250 and 200 L/hr with gas flow rate 100, 75 and 50 m³/hr and composition of CO_2 dissolved in water. The experiment had been run twice at different condition; with the present and absent of zeolites.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Over the past century, the Earth has increased in temperature by about 5 °C and many scientists believe this is because of an increase in concentration of the main greenhouse gases; carbon dioxide, methane, nitrous oxide, and fluorocarbons. The green house effect is the heating of the Earth due to the presence of greenhouse gases. It is named this way because of a similar effect produced by the glass panes of a greenhouse. Shorter-wavelength solar radiation from the sun passes through Earth's atmosphere and then is absorbed by the surface of the Earth causing it to warm as in Figure 2.1.



Figure 2.1: The process of global warming and how greenhouse gases create the greenhouse effect. (M.A.L.Caetano, 2008)

Carbon dioxide (CO_2) is a colorless, odorless non-flammable gas and is the most prominent Greenhouse gas in Earth's atmosphere as shown in Figure 2.2. It is recycled through the atmosphere by the process photosynthesis which makes human life possible.



Figure 2.2: The distribution of GHG in Earth's atmosphere. (Nordhaus, 1991)

Deforestation is the main producer of carbon dioxide. The causes of deforestation are logging for lumber, pulpwood, and fuel wood. Also contributing to deforestation is clearing new land for farming and pastures used for animals. Fossil Fuels were created chiefly by the decay of plants from millions of years ago. They use coal, oil and natural gas to generate electricity, heat the homes, power the factories and run the cars. These fossil fuels contain carbon. When they are burned, they combine with oxygen to form carbon dioxide. (Nordhaus, 1991)

The World Energy Council reported that global carbon dioxide emissions from burning fossil fuels rose 12% between 1990 and 1995 (UNEP, 2007). The increase from developing countries was three times that from developed countries. Middle East carbon dioxide emissions from burning of fossil fuels increased 35%, Africa increased 12%, and Eastern Europe increased rates by 75% from 1990-1995. Figure 2.3 shows the increase of carbon dioxide in the air over the past few centuries.



Figure 2.3: The increase of carbon dioxide in the air (UNEP, 2007)

2.2 Fundamentals

2.2.1 Adsorption Principles

The potential theory of adsorption was first introduced by Polanyi (1914) and later modified by Dubinin (1915) for adsorption on micro porous adsorbents. The theories are still regarded as fundamentally sound and accepted as correct as and better than all the other theories. This longevity of the theory is due to its essentially thermodynamic character and lack of insistence on a detailed physical picture.

It is based on the idea that at the surface of the solid adsorbent, the adsorbed molecules of the gas or vapor are compressed by the forces of attraction acting from the surface to a distance into the surrounding space. Because the forces anchoring a molecule to the surface decay with distance, a multi molecular adsorbed film may be regarded as lying in an intermolecular potential gradient. The force of attraction at any given point in the adsorbed film can be conveniently measured by the adsorption potential, ε which is defined as the work done by the adsorption forces in bringing a molecule from the gas phase to that point. (Polanyi, 1914) concept of the cross-section for a typical gas-solid system can be represented as shown in Figure 2.4.



Figure 2.4: Schematic of the surface adsorbed layer according to potential theory. (Polanyi, 1914)

According to Weber and Van Vliet (1980), adsorption at a surface or interface is largely the result of binding forces between the individual atoms, ions, or molecules of an adsorbate and the surface. All of these forces having their origin in electromagnetic interactions. Four principle types of adsorption are exchange, physical, chemical and specific. In this work, physical and chemical adsorption will be focused.

- Physical adsorption results from the action of van der Walls forces, which are comprised of both London dispersion forces and classical electrostatic forces. An electric dipole consists of two equal and opposite charges separated by a distance. All molecules contain atoms composed of positively charged nuclei and negatively charged electrons. When a molecule is placed in an electric field between two charged plates, the field attracts the positive nuclei toward the negative plate and the electrons toward the positive plate. This electrical distortion or polarization of the molecule creates an electric dipole. When the field is removed, the distortion disappears, and the molecule reverts to its original condition. This electrical distortion of the molecule is called induced polarization; the dipole formed is an induced dipole.
- Chemical adsorption involves a reaction between an adsorbate and an adsorbent resulting in a change in the chemical form of the adsorbate. It involves exchange or sharing of electrons between the adsorbate molecules

and the surface of the adsorbent. The bond formed between the adsorbate and the adsorbent is essentially a chemical bond and is thus much stronger than in physical adsorption.

Often the amount adsorbed is measured as a function of the partial pressure or concentration at a given temperature and the result expressed as an *adsorption isotherm* (for a given adsorbate-adsorbent system, the equilibrium amount adsorbed is a function of pressure at constant temperature). The adsorption isotherm is the most extensively employed method for representing the equilibrium states of an adsorption system. It can give useful information regarding the adsorbate, the adsorbent and the adsorption process. It helps in the determination of the surface area of the adsorbent, the volume of the pores, their size distribution, the heat of adsorption and the relative absorbability of a gas or a vapor on a given adsorbent. There are three possible theoretical approaches for deriving adsorption isotherms; the kinetic approach, the statistical approach and the thermodynamic approach. There are many empirical adsorption models, but the most common is the Langmuir adsorption isotherm in its usual form is based on the following assumptions:

- (i) The adsorbed molecule or atom is held at definite, localized sites.
- (ii) Each site can accommodate one and only one molecule or atom.
- (iii) The energy of adsorption is a constant over all sites, and there is no interaction between neighboring adsorbates.



Figure 2.5: Langmuir adsorption isotherm. (Ralph T. Yang, 1997)

2.2.2 Absorption Principles

The term gas absorption conventionally refers to the intimate contacting of a gas or mixture of gases with a liquid so that one or more components of the gas mixture will dissolve in the liquid. As the gas feed is a mixture, what is normally required is the selective absorption of one component, as in the removal of nitrogen dioxide from waste stack gas.

When the concentration of the soluble gas in the gas phase has been reduced sufficiently or the concentration of the dissolved gas (solute) in the liquid phase has reached the required level, then two streams must be parted, thereby effecting a separation, if required. However, given the density difference between the gas and liquid phases, this is not normally a problem and special equipment is not normally required for this. (Roman Zarzycki, 1993)

The first really effective treatment of the gas absorption was the two film theory proposed by (Whitman, 1923). Although numerous other theories have been proposed since this treatment was presented, it has the advantages of relative simplicity and direct application to practical gas absorber design and as a consequence it is still widely used as the basis for design calculations. Although its application is restricted, its limitations are now well known and due allowance can be made for them so that safety factors (e.g. over sizing) can be built into designs produced from calculations based on this theory.

Gas absorption involves of mass transfer across a phase (gas/liquid) boundary (or interface) and it is possible to treat the overall process in three stages; transfer of gas molecules within the bulk gas phase towards the phase boundary, transfer of gas molecules across the phase boundary and their dissolution in the liquid phase at the boundary, and transfer of dissolved gas molecules (solute) within the bulk liquid phase away from the boundary.

In each of the three stages, gas molecules will be transferred by a combination of diffusion or convection. For a flowing system which is the general rule for gas absorption, transfer within each of the bulk phases is effectively due to overall mixing of that phase rather than to diffusion. Thus, for practical purposes, both the bulk phases may be considered to be well-mixed and essentially uniform concentrations are maintained within the gas and within the liquid, though obviously a concentration difference exists between the two phases. (Roman Zarzycki, 1993)

However, near the boundary or interface between the two phases, turbulence is reduced by viscous and frictional drag forces until at the boundary itself. There is thought to be a stationary bi-layer, i.e. two thin films, one gas and one liquid which are effectively at rest with respect to each other. Transport across these films will be by molecular diffusion, so that it is the films which provide the restriction or resistance to absorption, since transport by molecular diffusion in the films will inevitably be slower than transport by turbulent mixing in the bulk phases. (Roman Zarzycki, 1993)

In gas absorption, it involves at least three components. A gaseous stream containing the component to be removed flows upward through a packed tower while a fluid stream flows downward through the tower. The component that is to be removed crosses the phase boundary. The concentration gradient of the transfer component between the two streams causes mass transfer to occur. The type of packing will have an effect on the amount of CO_2 absorbed into water.

The most important attributes of an adsorbent for any application are: capacity, selectivity, kinetics, compatibility and cost. Adsorption capacity is the most important characteristic of an adsorbent. It is the amount of adsorbate taken up by the adsorbent, per unit mass or volume of the adsorbent. It depends on the fluid phase concentration, temperature and others conditions (especially the initial condition of the adsorbent).

Selectivity is related to capacity. The simplest is the ratio of the capacity of one component to that of another at a given fluid concentration. The ratio generally approaches a constant value as concentration drops toward.

All cyclic adsorption applications rely on regenerability, so that the adsorbent can operate in sequential cycles with uniform performance. This means each absorbable component (adsorptive or adsorbate) must be relatively weakly adsorbed. The heat of adsorption provides a measure of the energy required for regeneration and in that regard low values are desirable. Regeneration might be accomplished by thermal swing, pressure swing, chemical (by displacement, elution or supercritical extraction), or sometimes by a combination of those. Displacement would involve introducing a species that adsorbs more strongly than the adsorbate of interest while elution would entail dissolving the adsorbed material by a solvent that is weakly adsorbed it at all. In some cases, regeneration takes place by contacting the adsorbent with a fluid in another phase than is used during loading. This requires draining or displacement which might be time-consuming, so it is avoided whenever possible. The regenerability of an adsorbent affects the fraction of the original capacity that is retained (sometimes called the working capacity), time and energy. (Susianto, 2005)

Mass transfer kinetics is a catch-all term related to intraparticle mass transfer resistance. It is important because it controls the cycle time of a fixed bed adsorption process. Fast kinetics provides a sharp breakthrough curve while slow kinetics yields a distended breakthrough curve. The effect of a distended breakthrough curve can be overcome by adding adsorbent at the product end, or by increasing the cycle time (which reduces the throughput per unit of adsorbent). Both of these options affect the amount of adsorbent required in that the longer the cycle time, the greater the adsorbent inventory. Slow diffusion of any adsorbate is a disadvantage. To compensate for slow diffusion, it is also possible to use small particles but there is a corresponding sacrifice due to increase pressure drop. The common solution to that dilemma is to use relatively large particles and to employ on extra increment of adsorbent.

Compatibility covers various possible modes of chemical and physical attack that could reduce the life expectancy of the adsorbent such as attrition. For example, the adsorbent, binder and surface groups (depending on the type of adsorbent) should be inert to the carrier or solvent and should not irreversibly react with (or chemisorbs) the adsorbates. Likewise, operating conditions such as velocity, temperature, pressure and vibration should not cause undue disintegration of the adsorbent particles. (Susianto, 2005)

Cost is perhaps the most subtle characteristic to understand because it may vary from week to week, and sales rep to sales rep even for the same exact material.

2.2.3 Adsorbents

2.2.3.1 Types of Adsorbents

Adsorption of gas molecules using activated carbon is dominated by the van der Walls forces (i.e., dispersion and repulsion forces). The electrical charges on activated carbon are too weak (compared with other sorbents) or the positive and negative charges are too close to each other to exert any significant electric field or field gradient on the surface. In addition, the small pore sizes and the large surface area of activated carbon play the major role in gas adsorption. Consequently, the surface groups have significant effects only for adsorption of polar gases. The most extensively studied gas adsorbate molecule is water. The unique surface property gives activated carbon more advantages. It is the only commercial sorbent used to perform separation and purification processes without requiring prior stringent moisture removal, such as is needed in air purification. (W. John Thomas, 1998)

For the same reason, it is also widely used as a sorbent for processes treating aqueous solutions. Beside that, because of its large, accessible internal surface (and large pore volume), it adsorbs more nonpolar and weakly polar organic molecules than other sorbents do. Lastly, the heat of adsorption or bond strength is generally lower on activated carbon than on other sorbents. This is because only non-specific; van der Walls forces are available as the main forces for adsorption. Consequently, stripping of the adsorbed molecules is relatively easier and results in relatively lower energy requirement for regeneration of the sorbent. (W. John Thomas, 1998). Table 2.1 below shows the applications of sorbent to major industries.

Table2.1: Applications of Major Industrial Sorbents

Sorbent	Major Uses for Gas Separation	
Activated carbon	Removal of nonpolar gases and organic vapors, H2	
	purification	
Zeolites	Drying; H2 purification; air purification; air	
	separation; separation based on molecular size and	
	shape (e.g., aromatics);	
Silica gel	Drying; gas chromatography	
Activated alumina	Drying; gas chromatography	

2.2.3.1.1 Activated Carbon

Activated carbon is one of the most complex solids but it is the most versatile because of its extremely high surface area and micropore volume. Moreover, its bimodal pore size distribution provides good access of sorbate molecules to the interior. The structure of activated is complex and it is basically composed of an amorphous structure and a graphite- like microcrystalline structure. (Sungyee Lee, 2006) The graphitic structure is important from the capacity point of view as it provides "space" in the form of slit-shaped channel to accommodate molecules. Because of the slit shape the micropore size for activated carbon is reported as the micropore half-width rather than radius as in the case of alumina or silica gel. The arrangement of carbon atoms in the graphitic structure is similar to that of pure graphite. The layers are composed of condensed regular hexagonal rings and two adjacent layers are separated with a spacing of 0.335nm. The distance between two adjacent carbon atoms on a layer is 0.142nm (Ralph T. Yang, 2003).

The graphitic unit in activated carbon usually is composed of about six to seven layers and the average diameter of each unit is about 10nm. The size of the unit can increase under the action of graphitization and this is usually done at very high temperature (>1000°C) and in an inert atmosphere. The linkage between graphite units

is possible with strong cross linking. The interspaces between those graphite units will form pore network and its size is usually in the range of mesopore and macropore.

Macropore having a size range of greater than 100nm is normally not filled with adsorbate by capillary condensation (except when the reduced pressure is approaching unity). The volume of macropore is usually in the order of 0.2-0.5 cc/g and the area contributed by the macropore is usually very small, of the order of 0.5 m^2/g , which is negligible compared to the area contributed by the micropore. Macropores therefore are of no significance in terms of adsorption capacity but they act as transport pores to allow adsorbate molecules to diffuse from the bulk into the particle interior.

Mesopore also called transitional pores has a size range from 2nm to 10nm, and it is readily filled during the region of capillary condensation (P/P₀ > 0.3). The volume of mesopore is usually in the range of 0.1to 0.4cc/g and the surface area is in the range of 10-100 m²/g. mesopore contributes marginally to the capacity at low pressure and significantly in the region of capillary condensation. Like macropores, mesopores act as transport pore when capillary condensation is absent and they act as

conduit for condensate flow in the capillary condensation region. (R.C. Bansal, 2005).

Micropores are pores having size less than 2nm. These pores are slit-shaped and because of their high dispersive force acting on adsorbate molecule they provide space for storing most of adsorbed molecules and the mechanism of adsorption is via the process of volume filling.

According to Dubinin and Zaverina (1916), a microporous active carbon is produced when the degree of burn-off is less than 50% and a macroporous active carbon when the extent of burn-off is greater than 75%. When the degree of burn-off is between 50 and 75%, the product has a mixed porous structure and contains all types of pores. Dubinin proposed a classification of the pores that has now been adopted by the International Union of Pure and Applied Chemistry (IUPAC). This classification is based on their width (w), which represents the distance between the walls of a slit-shaped pore or the radius of a cylindrical pore.

Activated carbons comprise elementary microcrystallites stacked in random orientation and are made by the thermal decomposition of various carbonaceous materials followed by an activation process. Raw materials include hard and soft woods, rice hulls, refinery residuals, peat, lignin, coals, coal tars, pitches, carbon black and nutshell, such as coconut. Activated carbon cloths are made from cellulose-based woven cloth and can have a higher capacity and better kinetic properties than the granular, but cheaper forms. Cloths can have both high external surface areas and high internal surface areas. Activated carbons contain a full range of pore sizes. Micropore diameters are generally less than 2nm while macropore diameters are generally greater than 50nm. Some pores maybe inaccessible because they are closed at both ends. Control of the pore sizes and of their distribution in the manufacturing process allows a broad range of adsorbents to be available offering widely differing selectivities. Carbons for gas phase applications require smaller pores while carbons for liquid phase applications tend to have larger pore diameters, of the order of 3nm or larger. Carbons for liquid phase applications also need to be made with surfaces of the appropriate wet ability. (Carl L. Yaws, 1999)

The surface of an activated carbon adsorbent is essentially non-polar but surface oxidation may cause some slight polarity to occur. Surface oxidation can be created, if required, by heating in air at around 300°C or by chemical treatment with nitric acid or hydrogen peroxide. This can create some hydrophilic character which can be used to advantage in the adsorption of polar molecules but can cause difficulties in other applications such as the adsorption of organic compounds from humid gas streams. In general, however, activated carbons are hydrophobic and organophilic and therefore they are used extensively for adsorbing compounds of low polarity in water treatment, decolourization, solvent recovery and air purification applications. One advantage of activated carbon is that the adsorption of organic molecules tends to be non-specific.

Studies also indicate that macroporosity of the carbon affects transfer of adsorbate molecules to adsorption sites within the particle and that adsorption capacity and rate of adsorption depend not only upon the internal surface area, pore size distribution, and pore shape but also the surface chemistry of the activated carbon.

The interaction of free radicals on the carbon surface with atoms such as nitrogen and oxygen both from the carbon precursor and from the atmosphere result in the formation of functional groups when the carbon is being activated. These functional groups cause the surface of the activated carbon to become chemically reactive which influences the adsorptive properties of the activated carbon. As a result, the surface characteristics of the activated carbon can be amphoteric, that is either acidic or basic due to the formation of carboxylic groups, hydroxyl or carbonyl groups. These groups can interact with water causing increased hydrophilicity of the carbon surface. The formations of these groups influence the electrokinetic properties, and affect not only use as a catalyst support, and the adsorption of various molecular species, but also the reaction with certain solvents in solvent recovery applications. (Bu Guo, 2006)

Other important properties of activated carbon include particle size distribution and pore volume. Particle size distribution can generally be indicated by plotting a range of pore diameters against pore volume or pore surface area. Gasadsorbing carbons usually have a high volume of pores with diameters of less than about 30 to about 32 Angstroms and a low volume of pores having diameters greater than about 30 to about 32 Angstroms up to about 2000 to about 4000 Angstroms, whereas activated carbons employed in liquid-phase separations have a significant volume of pores in the latter range. (E. Alvarez, 2007).

This larger pore diameter range improves the accessibility of larger molecules thereby facilitating adsorption of larger molecules as well as colloidal structures. Powdered or granular forms of activated carbons find use in liquid-phase separation, whereas all gas- and vapor-phase adsorption processes employ granular carbon. Pelletized, extruded fiber and impregnated forms of activated carbons also find use in separation processes in addition to the powdered or granular forms (Norit, 1998).

From the previous research, the volume adsorbed on the carbon samples decreased with increasing temperature in all cases. The volume adsorbed on the carbons does not exhibit any relationship with the physical properties such as BET surface area and pore volume. (Bo Guo, 2006)

2.2.3.1.2 Zeolites

Zeolites are microporous crystalline solids with well-defined structures. Generally they contain silicon, aluminium and oxygen in their framework and cations, water or other molecules within their pores. Many occur naturally as minerals and are extensively mined in many parts of the world. Others are synthetic and are made commercially for specific use or produced by research scientists trying to understand more about their chemistry. (K.S.Nor, 2005)

Because of their unique porous properties, zeolites are used in a variety of applications with a global market of several million tonnes per annum. In the western world, major uses are in petrochemical cracking, ion-exchange (water softening and purification) and in the separation and removal of gases and solvents. Other applications are in agriculture, animal husbandry and construction. They are often also referred to as *molecular sieves*. (Z.Zhao, 2007).

A defining feature of zeolites is that their frameworks are made up of 4 connected networks of atoms as shown in Figure 2.6. This is in terms of tetrahedral, with a silicon atom in the middle and oxygen atoms at the corners. These tetrahedral can then link together by their corners to from a rich variety of beautiful structures. The framework structure may contain linked cages, cavities or channels which are of the right size to allow small molecules to enter the limiting pore sizes between 3 and 10 Å in diameter. In all, over 130 different framework structures are now known. In addition to having silicon or aluminium as the tetrahedral atom, other compositions have also been synthesized including the growing category of microporousaluminophosphates, known as ALPOs.



Figure 2.6: Framework structure (R.Li, 2007)

The shape-selective properties of zeolites are also the basis for their use in molecular adsorption. The ability preferentially to adsorb certain molecules, while excluding others has opened up a wide range of molecular sieving applications. Sometimes it is simply a matter of the size and shape of pores controlling access into the zeolite. In other cases different types of molecule enter the zeolite, but some diffuse through the channels more quickly, leaving others stuck behind as in the purification of *para*-xylene by silicalite. Cation-containing zeolites are extensively used as desiccants due to their high affinity for water and also find application in gas separation; molecules are differentiated on the basis of their electrostatic interactions

with the metal ions. Conversely, hydrophobic silica zeolites preferentially absorb organic solvents. Zeolites can thus separate molecules based on differences of size, shape and polarity. (Z.Zhao, 2007).

CHAPTER 3

METHODOLOGY

3.1 Introduction

Gas adsorption - absorption on activated carbon and zeolites give the basis for a variety of processes such as gas separation and purification, adsorptive natural gas storage and hydrogen gas storage. These processes are more attractive since they are economical and could control air pollution problem today. Therefore, there is a real need to evaluate the potential between adsorption and absorption process. Because of that, this experiment had been conducted by using Gas Absorption-Adsorption Unit. In order to achieve the objective and scope of this study, this chapter presents the methods and materials used throughout of the study. The experimental procedure in this study involves gas adsorption measurement. The gas adsorption measurements involved determination of gas composition and study the effect of gas and liquid flow rate.

3.2 Methodology Procedure

In order to achieve the objective of this study, a few steps need to be taken and consider for running the experiment. Figure 3.1 below shows the flow diagram of methodology adopted in this study.



Figure 3.1: Methodology Flow Chart

3.3 Materials

In order to examine the effect of CO_2 gas removal, the experiment had been run with and without gas flow through the zeolites. The zeolites were purchased from Fischer Scientific (M) Sdn Bhd, Shah Alam Selangor.

 Table 3.1: Properties of Zeolites

Form	Granular
Composition	Ag, 35%
Particle size	+20 mesh
mp	>500°C (lit)
Density	1.07 g/mL at 25°C (lit)
Molecular formula	$Ag_{84}Na_{2}[(AlO_{2})_{86}(SiO_{2})_{106}] \cdot xH_{2}O$

3.4 Carbon Dioxide Gas

Regarding on the scope of this study which is to improve adsorption absorption ability of CO₂ gas, a cylinder of Purified Carbon Dioxide was purchased from MOX Malaysia Petaling Jaya, Selangor.

Table 3.2: Properties of Carbon Dioxide

Molecular formula	CO ₂
Molar mass	44 g/mol
Melting point	-57°C (216 K) (under pressure)
Boiling point	−78 °C (195 K)
Solubility in water	1.45 g/L
Appearance	Colorless
Viscosity	0.07 cP at -78 °C
Dipole moment	zero

For the portable tube of zeolite, the vinyl tube, T-connector and needle valve were purchased from Eng Bee Brothers Hardware Trading Sdn Bhd Kuantan, Pahang.

Figure 3.2 above shown the equipments needed.



Figure 3.2: PFA Tubing and Needle Valve

3.6 Experimental Work

3.6.1 The absorption-adsorption process using Gas Absorption-Adsorption Unit

The portable tube of zeolites is connected to V3. The feed vessel B1 is fill with water and start with packed column K1. The valve V8 is open. Circulation pump P1 is switch on and then valve V12 and V11 is open and adjust to give a water flow rate 250 L/hr. The water is allow entering the top of column K1, flowing down the column and accumulate at the bottom until it overflows back into feed vessel B1. Valve V3 is open and adjust to give a flow rate of $100m^3/hr CO_2$ into column K1. The gas mixture is allowed to flow through the packed column for 10 minutes. The liquid samples are start collecting from B1 inlet and outlet of the packed column K1. The samples always collect simultaneously. The collected samples are analyzed to determine the composition of dissolved CO_2 .

- i. 100 ml of sample is preparing in a conical flask.
- 5-10 drops of phenolphthalein indicator is adding. If the sample turns red immediately, no free CO₂ is present. If the samples remain colorless, titrate with standard 0.05 M NaOH solution.
- iii. The volume of 0.05 M NaOH required to reach the end point is measured. The end point occurs when a definite pink color persists in the solution for longer than 30 seconds.

The liquid samples is continue to collect at every 5 minutes intervals. Throughout the experiment, the CO_2 and water flow rates is monitor and maintain by adjusting the appropriate valves. The experiment is continued with a water flow rate 200 L/hr at constant gas flow rate. The experiment is repeated for constant water flow rate at 250m³/hr with gas flow rate is 75 and 50m³/hr. In order to study the effect of CO_2 gas removal, the experiment is repeated without the portable tube of zeolites. At the end of the experiment, valve V4 is closed and circulation pump P1 is switch off. The liquid from the bottom of column K1 is drain by operating valve V6. All liquid from the feed vessel B1 is drain by opening valve V10. The system is shutting down. Figure 3.2 shows the PFD of Gas Adsorption-Absorption Unit before connect to portable tube of zeolites.



Figure 3.3: PFD of Gas Adsorption-Absorption Unit

CHAPTER 4

RESULT AND DISCUSSION

In this study, the preparation of 0.05M NaOH solution is shown in Appendix A while the percentage of CO_2 for each sample is shown in Appendix B.

4.1 Data for Zeolites (present)

4.1.1 Gas Flow Rate Constant (100m³/hr)

At the condition 250 L/hr and 200 L/hr of water flow rate with constant gas flow rate, Table 4.1 and Table 4.2 are tabulated as in Appendix C. From that, the graph is plotted as in Figure 4.1.

Figure 4.1 below shows the graph of CO_2 composition versus time for constant gas flow rate. The figure illustrates the changes of CO_2 composition with water flow rate at 250 and 200 L/hr. From the graph, it shows that increasing the water flow rate will increase the composition of CO_2 dissolved in water solution. The increasing of CO_2 composition for the volume of feed will decrease the efficiency of the process since the desired product is still mixed at the feed vessel. This shows that increase.



Figure 4.1: Graph of CO₂ composition vs. time for constant gas flow rate

4.1.2 Water Flow Rate Constant (250L/hr)

At the condition of $75m^3/hr$ and $50m^3/hr$ of gas flow rate with constant water flow rate, Table 4.3 and Table 4.4 are tabulated as in Appendix C. From that, the graph is plotted as in Figure 4.2.

Figure 4.2 below shows the graph of CO_2 composition versus time for constant water flow rate. The figure illustrates the changes of CO_2 composition with gas flow rate at 75 and 50m³/hr. CO_2 composition decrease with time. From the graph, it shows that increasing the gas flow rate will decrease the composition of

 CO_2 dissolved in water solution. This shows that increasing the gas flow rate will decrease the rate of absorption of CO_2 in water solution.



Figure 4.2: Graph of CO₂ composition vs. time for constant water flow rate

4.2 Data for Zeolites (absent)

4.2.1 Gas Flow Rate Constant (100m³/hr)

At the condition 250 L/hr and 200 L/hr of water flow rate with constant gas flow rate, Table 4.5 and Table 4.6 are tabulated as in Appendix D. From that, the graph is plotted as in Figure 4.3.

Figure 4.3 below shows the graph of CO_2 composition versus time for constant gas flow rate. The figure illustrates the changes of CO_2 composition with water flow rate at 250 and 200 L/hr. From the graph, it shows that increasing the water flow rate will increase the composition of CO_2 dissolved in water solution.

This shows that increasing water flow rate causes the absorption of CO_2 in water increase. Beside that, the adsorption process only happens on activated carbon since there were no others adsorbent (zeolites). The CO_2 composition at the final is 0.0003950% bigger than previous experiment which is 0.0002872%. On the other hand, the CO_2 composition is bigger without zeolites.



Figure 4.3: Graph of CO₂ composition vs. time for constant gas flow rate

4.2.2 Water Flow Rate Constant (250L/hr)

At the condition of $75m^3/hr$ and $50m^3/hr$ of gas flow rate with constant water flow rate, Table 4.7 and Table 4.8 are tabulated as in Appendix D. From that, the graph is plotted as in Figure 4.4.

Figure 4.4 below shows the graph of CO_2 composition versus time for constant water flow rate. The figure illustrates the changes of CO_2 composition with gas flow rate at 75 and 50m³/hr. From the graph, it shows that increasing the gas

flow rate will increase the composition of CO_2 dissolved in water solution. In this case, it shows that increasing the gas flow rate will also increase the rate of absorption of CO_2 in water solution. Beside that, the adsorption process only happens on activated carbon since there were no others adsorbent (zeolites). The CO_2 composition at the final is 0.0006929% bigger than previous experiment which is 0.0001198%. On the other hand, the CO_2 composition is bigger without zeolites present.



Figure 4.4: Graph of CO₂ composition vs. time for constant water flow rate

The effect of removing the adsorbent (zeolites) can be seen clearly by plotting the graph such as in Figure 4.5 and Figure 4.6. These two figures are at the condition of constant gas flow rate, 100m³/hr.

Figure 4.5 and Figure 4.6 below show the graph of CO_2 composition versus time. The figure illustrates the changes of CO_2 composition with the present and absent of zeolites at different water flow rate. From the graph, it shows that the CO_2 composition increase with the present of zeolites. This might be cause of the gas

leakage between the connections of the gas cylinder with the tube of zeolites. Beside that, the water and gas flow rate at the panel is not maintained as had been set during this experiment.



Figure 4.5: Graph of CO₂ composition vs. time for 250L/hr water flow rate



Figure 4.6: Graph of CO₂ composition vs. time for 200L/hr water flow rate

Figure 4.7 and 4.8 show the effect of removing the adsorbent (zeolites) at the condition of constant water flow rate, 250L/hr.

Figure 4.7 and Figure 4.8 below show the graph of CO_2 composition versus time. The figure illustrates the changes of CO_2 composition with the present and absent of zeolites at different gas flow rate. From the graph, it shows that the CO_2 composition decrease with the present of zeolites. This shows that gas had been adsorbed twice along the process; at the zeolites and activated carbon. By having zeolites, it helps to decrease the rate of absorption of CO_2 .



Figure 4.7: Graph of CO₂ composition vs. time for 75m³/hr gas flow rate



Figure 4.8: Graph of CO₂ composition vs. time for 50m³/hr gas flow rate

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on the result obtained from the analysis, this study can be concluded where increasing the water flow rate will increase the composition of CO_2 dissolved in water solution and increasing the gas flow rate will reduce the composition of CO_2 dissolved in water solution. Beside that, the CO_2 composition decrease with the present of zeolites.

At the end of this study, all two objectives are achieved where the first objective; to compare the effect of changing the gas removal techniques is accomplished. Increasing the gas flow rate will decrease the CO_2 composition dissolved in water solution. Higher gas flow rate increases the efficiency of the process. This shows that increasing the gas flow rate will decrease the rate of CO_2 absorption in water as illustrated in Figure 4.2, Figure 4.4, Figure 4.7 and Figure 4.8.

On the other hand, increasing the water flow rate from 200 to 250L/hr will increase the CO₂ composition dissolved in water solution. This shows that increasing water flow rate causes the rate of CO₂ absorption in water increase. The increasing of CO₂ composition for the volume of feed will decrease the efficiency of the process since the desired product is still mixed at the feed vessel. Increasing of water flow rate will increase the CO₂ composition dissolved even greater rather than increasing gas flow rate. The CO_2 compositions at different water flow rate with constant gas flow rate are illustrated in Figure 4.1, Figure 4.3, Figure 4.5 and Figure 4.6.

The second objective is to study the performance on the CO_2 gas removal from different adsorbent. The CO_2 composition decrease when the zeolites are present. This shows that gas had been adsorbed twice along the process; at the zeolites and activated carbon. By having zeolites, it helps to decrease the rate of absorption of CO_2 . The CO_2 composition at different water and gas flow rate with the present or absent of zeolites are illustrated in Figure 4.5, Figure 4.6, Figure 4.7 and Figure 4.8.

5.2 Recommendation

From the study conducted, there are several recommendations for improving and furthering the study is as follow;

- I. The process will be more efficient if the CO₂ composition in the receiving vessel is high in order to have a long contact time between liquid and gas phase with the adsorbent.
- II. Use another type of gas such as NO₂ or SO₂ which are the polluted gaseous to have accurate composition of gas.

APPENDIX A

Preparation of 0.05M NaOH solution

The 0.05M NaOH solution in 1L volume is prepared:

Mass = Molar x Molecular weight = 0.05mol/L x 40g/mol = 2 g/L of NaOH

Stoichiometry equations:

$$CO_2 + H_2O \rightarrow H_2CO_3 \tag{4.1}$$

$$H_2CO_3 + 2NaOH \rightarrow Na_2CO_3 + 2H_2O \tag{4.2}$$

2 mol of NaOH react with 1 mol of H_2CO_3 Hence, mol of CO_2 equals to 1/2 mol of NaOH

Mol of NaOH = MV/1000 = (0.05) (0.2)/1000= $1.0 \ge 10^{-5}$ mol

From the equation 4.2, 1 mol of H₂CO₃ reacts with 2 mol of NaOH, so: No. of mol of H₂CO₃= 1.0×10^{-5} mol NaOH X 1 mol H₂CO₃ 2 mol NaOH = 5 x 10⁻⁶ mol H₂CO₃

From the equation 4.1, 1 mol of CO_2 produces 1 mol of H_2CO_3 , then:

No. of mol of CO₂ = $5 \times 10^{-6} \mod H_2 CO_3 \times 1 \mod CO_2$ 1 mol H₂CO₃ = $5 \times 10^{-6} \mod CO_2$

To find the mass of CO_2 , from the equation: mol = mass/MV

Mass of CO₂ = mol x molecular weight
= 5 x 10⁻⁶ mol CO₂ X 44g/mol
=
$$2.2 \times 10^{-4}$$
g

APPENDIX B

Percentage of CO₂

Percentage of CO₂ in the first sample:

$$= \frac{\text{mass of CO}_2}{\text{mass of sample}} X 100\%$$

mass of sample
$$= \frac{2.2 \times 10^{-4} \text{g X}}{186.2} X 100\%$$

186.2
$$= 0.0001182 \% \text{ of CO}_2 \text{ in first sample}$$

** NOTE THAT THE CALCULATION OF THE PERCENTAGE OF CO₂ IS ALL AS SAME AS ABOVE.

APPENDIX C

Data for Zeolites (present)

Table 4.1: CO_2 composition at water flow rate 250 L/hr and gas flow rate is $100m^3/hr$.

Time (min)	Sample mass (g)	Vol. of NaOH (ml)	% CO ₂ (outlet)
5	186.2	0.2	0.0001182
10	176.2	0.3	0.0001873
15	163.2	0.6	0.0004044
20	167.1	0.6	0.0003950

Table 4.2: CO_2 composition at water flow rate 200 L/hr and gas flow rate is $100m^3/hr$.

Time (min)	Sample mass (g)	Vol. of NaOH (ml)	% CO ₂ (outlet)
5	178.2	0.6	0.0003704
10	152.7	0.3	0.0002161
15	174.1	0.3	0.0001895
20	163.5	0.3	0.0002018

Table 4.3: CO₂ composition at gas flow rate 75m³/hr and water flow rate is 250L/hr.

Time (min)	Sample mass (g)	Vol. of NaOH (ml)	% CO ₂ (outlet)
5	185.3	0.4	0.0002375
10	201.0	0.4	0.0002189
15	188.1	0.6	0.0003509
20	183.7	0.2	0.0001198

Time (min)	Sample mass (g)	Vol. of NaOH (ml)	% CO ₂ (outlet)
5	194.7	0.6	0.0003390
10	183.0	0.4	0.0002404
15	184.3	0.2	0.0001194
20	181.0	0.2	0.0001215

Table 4.4: CO_2 composition at gas flow rate 50 m³/hr and water flow rate is 250L/hr.

Data for Zeolites (absent)

Table 4.5: CO_2 composition at water flow rate 250 L/hr and gas flow rate is $100m^3/hr$.

Time (min)	Sample mass (g)	Vol. of NaOH (ml)	% CO ₂ (outlet)
5	190.1	0.7	0.0004050
10	191.6	0.4	0.0002296
15	186.8	0.3	0.0001767
20	191.5	0.5	0.0002872

Table 4.6: CO₂ composition at water flow rate 200 L/hr and gas flow rate is $100 \text{m}^3/\text{hr}$.

Time (min)	Sample mass (g)	Vol. of NaOH (ml)	% CO ₂ (outlet)
5	197.3	0.3	0.0001673
10	182.4	1.0	0.0006030
15	184.8	0.3	0.0001786
20	171.5	0.3	0.0001924

Table 4.7: CO₂ composition at gas flow rate 75m³/hr and water flow rate is 250L/hr.

Time (min)	Sample mass (g)	Vol. of NaOH (ml)	% CO ₂ (outlet)
5	188.7	1.1	0.0006412
10	186.0	0.5	0.0002957
15	182.5	1.0	0.0006027
20	190.5	1.2	0.0006929

Table 4.8: CO₂ composition at gas flow rate 50 m³/hr and water flow rate is 250L/hr.

Time (min)	Sample mass (g)	Vol. of NaOH (ml)	% CO ₂ (outlet)
5	192.9	0.7	0.0003992
10	188.0	0.9	0.0005266
15	183.6	1.0	0.0005991
20	188.4	0.5	0.0002919

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