

**STUDY OF PURITY AND RECOVERY OF CARBON DIOXIDE  
FROM BINARY MIXTURE OF NATURAL GAS USING  
DIFFERENT ADSORBENTS IN PRESSURE EQUALIZED  
PRESSURE SWING ADSORPTION**

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**UNIVERSITI SAINS MALAYSIA**

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DIFFERENT ADSORBENTS IN PRESSURE EQUALIZED  
PRESSURE SWING ADSORPTION**

**by**

**CHONG CHING YANG**

**Thesis submitted in partial fulfilment of the requirement  
for the degree of Bachelor of Chemical Engineering**

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## LIST OF SYMBOL

$C$	Concentration	g/l
$C_0$	Initial concentration	g/l
$P/P_0$	Relative pressure	-

## LIST OF ABBREVIATION

CO <sub>2</sub>	Carbon Dioxide
CH <sub>4</sub>	Methane
PSA	Pressure Swing Adsorption
VPSA	Vacuum Pressure Swing Adsorption
CMS	Carbon Molecular Sieve
UiO-66	Zirconium-benzenedicarboxylate
MOF	Metal Organic Framework
AC	Activated Charcoal
EDX	Energy Dispersive X-Ray

**KAJIAN KETULENAN DAN PEMULIHAN KARBON DIOKSIDA  
DARIPADA CAMPURAN BINARI GAS ASLI DENGAN PENJERAP  
BERBEZA DALAM TEKANAN EKUALISASI  
PENJERAPAN TEKANAN BUAIAN**

**ABSTRAK**

Kesan pemisahan metana ( $\text{CH}_4$ ) dan carbon dioksida ( $\text{CO}_2$ ) merupakan campuran binari gas asli telah disiasat dengan ujian penyifatan, kajian proses penembusan dan penjerapan tekanan buaian (PSA). 2 jenis penjerap dikaji dan dibandingkan iaitu arang teraktif dan zirkonium-benzenedicarboxylate (UiO-66). Ujian penyifatan menunjukkan bahawa UiO-66 mempunyai luas permukaan BET yang lebih tinggi dan arang teraktif mempunyai kandungan karbon tetap yang lebih tinggi. Untuk kajian proses penembusan, keluk proses penembusan telah diperolehi dalam keadaan 1 bar dan suhu bilik. Kesan kedua-dua penjerap dan nisbah kemasukan gas yang berbeza telah disiasat. UiO-66 menunjukkan ia mempunyai kapasiti penjerapan yang lebih besar dibuktikan dengan masa yang lebih lama dalam kajian proses penembusan. Di samping itu, proses PSA telah menggunakan penjerapan 2 katil dalam keadaan 3 bar dan suhu bilik. Walau bagaimanapun, kesan pemisahan campuran ini tidak berkesan dan tertarik dari segi komersial kerana 2 jenis penjerap yang digunakan tidak sesuai untuk memisahkan binary gas ini. Oleh itu, ketulenan tidak tercapai tetapi pemulihan boleh diperolehi kerana UiO-66 menunjukkan lebih daripada 90% ke atas untuk pemulihan  $\text{CO}_2$ .

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**ABSTRACT**

The separation effect of methane (CH<sub>4</sub>) and carbon dioxide (CO<sub>2</sub>) which is binary mixture of natural gas was investigated with characterization tests, breakthrough study and pressure swing adsorption (PSA) process. 2 types of adsorbent were studied and compared which were activated charcoal and zirconium-benzenedicarboxylate (UiO-66). The characterization tests showed that UiO-66 has higher BET surface area and activated charcoal has higher fixed carbon content. For breakthrough study, the breakthrough curves were obtained under the condition of 1 bar and room temperature. The effects of both adsorbents and different inlet gas ratio were investigated. UiO-66 has larger adsorption capacity proven by its longer breakthrough time. Besides, PSA process had utilized 2-bed adsorption under the conditions of 3 bar and room temperature. However, separation effect of this mixture was not effective and commercially attractive because 2 types of adsorbent used were not suitable for separating this binary gas. Hence, the purity was not achieved but the recovery could be obtained because UiO-66 showed more than 90% recovery on CO<sub>2</sub>.

## CHAPTER ONE

### INTRODUCTION

#### 1.1 Research Background

The concentration of carbon dioxide (CO<sub>2</sub>) and the main source of CO<sub>2</sub> emissions is from the combustion of fossil fuels such as natural gas in the atmosphere continue to increase. There are some serious concerns about the effects of CO<sub>2</sub> on the environment have been raised and there are considered to be the main anthropogenic contributor to the greenhouse gas effect and are responsible for 60% of the observed increase in the temperature of the atmosphere, which is commonly referred to as global warming (Yua et al., 2012). Pressure swing adsorption has become very prominent in the purification of gases for multiple applications, namely air purification (Rege et al., 2001; Hassan et al., 1986), hydrogen separation and purification (Malek and Farooq, 1998) and CO<sub>2</sub> capture (García et al., 2013; Pirngruber and Leinekugel-le-Cocq, 2013). The system is a popular technique, a reliable and economic on-site supply method. Many types of porous media have been used for CO<sub>2</sub>/CH<sub>4</sub> separation such as carbon molecular sieves (Bai et al., 2013), zeolites (Cavenati et al., 2004), metal organic framework (Barcia et al., 2008), and activated carbon (Goetz et al., 2006).

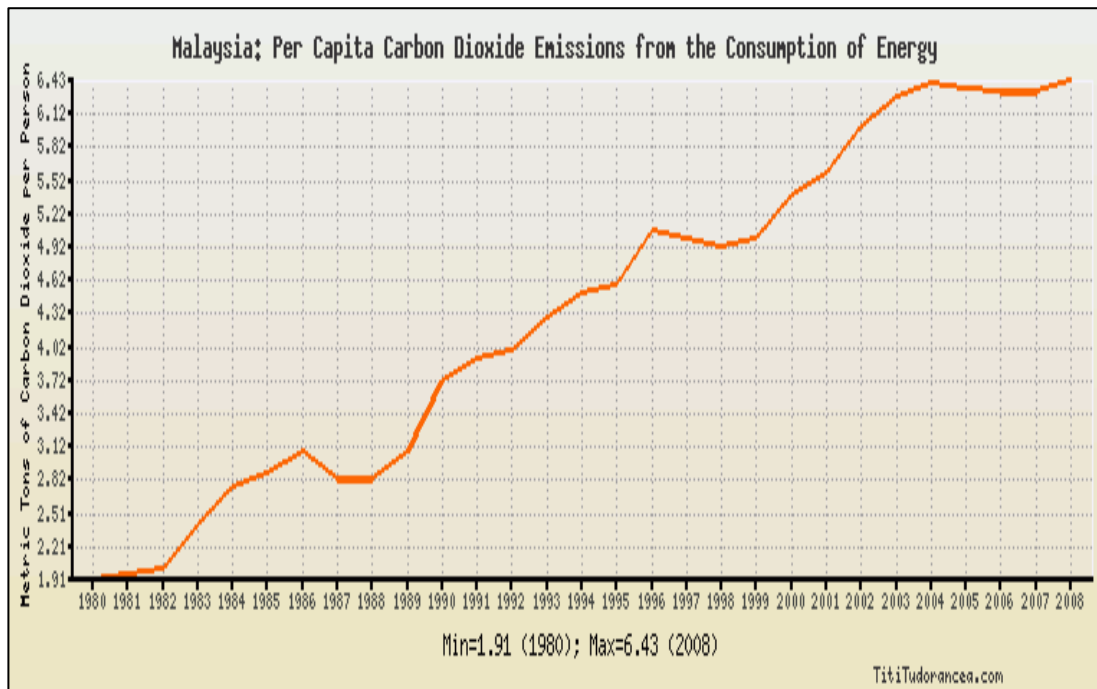


Figure 1.1: The chart shows CO<sub>2</sub> emissions (per capita) from the consumption of energy in Malaysia (Business, 2011).

According to the Figure 1.1, the largest source of carbon dioxide emissions is from the production of energy which is also combustion of fossil fuel. Emission of carbon dioxide from combustion of fossil fuel stands 87% of total human carbon dioxide emissions. In year 2011, fossil fuel created 33.2 billion tonnes of carbon dioxide emissions worldwide (Qu  r   et al., 2012). The 3 main types of fossil fuels are coal natural gas and oil. Coal is responsible for 43% of carbon dioxide emissions from fuel combustion, 36% is produced by oil while the remaining 20% belongs to natural gas as shown in Figure 1.2 (Defra, 2014). Hence, the study CO<sub>2</sub> recovery and purity from binary natural gas is essential to tackle the massive carbon dioxide emission.

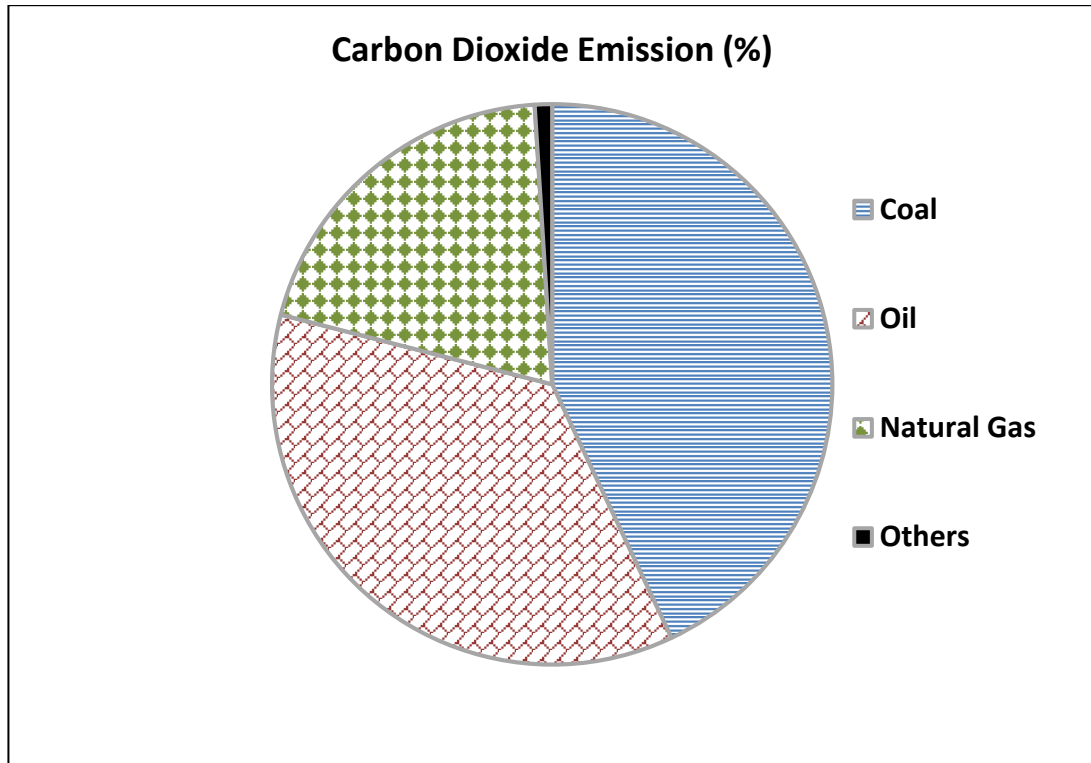


Figure 1.2: The pie chart shows the percentage of CO<sub>2</sub> emissions from different non-renewable energy sources (Defra, 2014).

Therefore, the focus of the present study is to study the purity and recovery of CO<sub>2</sub> from binary mixture of natural gas through pressure swing adsorption (PSA) using different adsorbents.

## 1.2 Problem Statement

The main composition in binary mixture of natural gas is CO<sub>2</sub> and CH<sub>4</sub>. Current researchers most likely focus on the ways or strategies to enrich the natural gas in order to increase the market value of it. Hence, CO<sub>2</sub> level in natural gas has to be reduced to enrich the natural gas and minimize the emission of CO<sub>2</sub> during combustion of fuel. Besides, CO<sub>2</sub> has an acidic nature and it causes corrosion in piping while it forms



carbonic acid when it reacts with water. Therefore, separation of CO<sub>2</sub> and CH<sub>4</sub> is the main focus of this study.

For current technology, the most common method to separate binary mixture of natural gas is through cryogenic distillation. The cryogenic method is better at extraction of the lighter liquids, such as methane than other alternative absorption method (Tobin et al., 2006). However, CO<sub>2</sub> is only a byproduct from natural gas processing. Most of the researches focusing in producing the highest purity of natural gas because of the market demand.

According to an equipment supplier from India, Table 1.1 shows recovery details from different kind of gases.

Table 1.1: Various Pressure Swing Adsorption (PSA) / Vacuum Pressure Swing Adsorption (VPSA) processes offered for gas separation and purification (Ltd, 2016).

	<b>Nitrogen</b>	<b>Hydrogen</b>	<b>Methane</b>	<b>Carbon Dioxide</b>
<b>Process</b>	PSA	PSA	VPSA PSA	VPSA PSA
<b>Feed</b>	Air N <sub>2</sub> -off gas	H <sub>2</sub> -off gas Natural gas Methanol NH <sub>3</sub>	Biogas Mine gas Natural gas	CO <sub>2</sub> -off gas Flue gas
<b>Product Purity</b>	95-99.99%	> 99.99%	> 98%	90/98%
<b>Flow (Nm<sub>3</sub>/h)</b>	1-3	< -30,000	< 10,000	< 10,000
<b>Adsorbent</b>	CMS	CMS Zeolite AC	CMS AC	CMS Zeolite AC

The current situation for CO<sub>2</sub> recovery from natural gas requires more research and studies especially on PSA or VPSA processes. Different adsorbents are encouraged to study on its separation and purification performance on natural gas processing.

Therefore, PSA and VPSA studies are introduced to this research in order to find alternative methods to recover the highest amount and purity of CO<sub>2</sub>. The expected result for this research depends on the types of adsorbents. However, the predicted recovery and purity of CO<sub>2</sub> in overall are both at least 80% as from the information above the purity of gases recovered is very high.

### **1.3 Research Objectives**

The objectives of the present research are:

1. To prepare, characterize, and analyse the adsorbents used for removal of CO<sub>2</sub> through PSA process.
2. To analyse the breakthrough study on adsorption of CO<sub>2</sub>/CH<sub>4</sub> from binary mixture of natural gas by varying the operating parameter such as gas ratio and adsorbent types.
3. To study the performance of PSA in term of purity and recovery by manipulating initial pressure, gas ratio and types of adsorbent.

## **1.4 Scope of Study**

Adsorbent is one of the crucial element in this research because adsorbent surface adsorbs gas particles on its surface. Before preparation starts, the raw materials and chemicals are collected or ordered from suppliers. Method of preparations are searched and decided. Different types of adsorbents are then prepared to for experiment use. After the preparation phase, characterization of adsorbent provides the properties and details of each adsorbent.

In this research, several adsorbents are selected to undergo breakthrough of CO<sub>2</sub> and CH<sub>4</sub> which is binary mixture of natural gas. During breakthrough process, the separation performance of each adsorbent with different binary gas composition is investigated. The time of breakthrough for adsorbents is taken as reference for upcoming PSA process.

For PSA process, pressure is manipulated to study the best pressure by using different type of adsorbents. This PSA is a 2-bed process which involves pressurization, depressurization, purging and equalization. These steps are manipulated by adjusting the valves in the rig setup.

## **1.5 Organization of Thesis**

This research paper consists of total 5 chapters. Chapter 1 writes up about the background and purpose of the research paper. The objectives and problem statement are also well explained in this section. For more detailed information, Chapter 2 covers related theory and journal studies about adsorbents, breakthrough and equalized pressure PSA. The effect of parameters to PSA and breakthrough are listed in second

chapter. All of the material used for this whole research and procedure of experiments are included in Chapter 3.

Chapter 4 describes the experimental results and discussions of the data obtained. Further elaboration on the effect of different adsorbent and parameters on the breakthrough and PSA process. Chapter 5 is the last chapter and it concludes all the findings obtained in this study. Recommendations are also included in the last chapter as well.

## CHAPTER TWO

### LITERATURE REVIEW

This literature review mainly writes up about the adsorption, adsorbents, breakthrough process and also PSA. It contains all of the related information extracted from other researches that contribute to this research.

#### 2.1 Natural Gas

Natural gas is a fossil fuel and its combustion normally causes global warming emission. Natural gas is a mixture of different gases but the main composition is CH<sub>4</sub> (Saidur et al., 2007). The higher CH<sub>4</sub> content in natural gas, the higher market value it gets. However, the composition of natural gas is not constant because it may contain other components such as ethane, propane and butane as well as nitrogen, helium, carbon dioxide, hydrogen sulphide and even water content (DeLuchi et al., 1988). Natural gas is one of the cleaner fossil fuels and its reserves are 5288.5 trillion cubic feet (Energy, 2002). It is easy to transport and store, available on demand, and cheaper than gasoline and diesel (Jaramillo et al., 2007). It is cheap and available in great quantities in many parts of the world. CO<sub>2</sub> emitted from every 1000MJ of energy released from combustion of natural gas is 53.07 kg CO<sub>2</sub>. Hence, it is much lesser comparing with other fossil fuel (Administration, 2016).

## **2.2 Adsorption**

In the previous couple of decades, many techniques had been utilized to study and recover the highest purity of CO<sub>2</sub>. The more common methods were adsorption (Chapel et al., 1999; Mimura et al., 1997) and membrane filtration (Favre, 2007).

Adsorption is defined as adhesion of a chemical species onto a surface of particles. Adsorption is a common separation technique. During the adsorption process, the gas or liquid (adsorbate) bind to a solid or liquid surface (adsorbent). The adsorption works depend on the surface energy. When the atoms on adsorbent surface are partly exposed, adsorbate molecules are attracted to the surface atoms. Adsorption may result from chemisorption, physisorption and electrostatic attraction (Education, 2017). Chemisorption is a kind of adsorption which the adsorbed substances is held by chemical bonds. Physisorption is also called physical adsorption is a process in which atoms or molecule is barely attached on the surface of adsorbent.

Adsorption isotherms are also used to describe adsorption because different parameters will cause different effects on the process. Several models have been developed to describe adsorption which are linear, Langmiur, Freundlich, Brunauer–Emmett–Teller, Redlich–Peterson and others (Foo and Hameed, 2010).

### **2.2.1 Pressure Swing Adsorption (PSA)**

The separation technique between 2 gases is very important for current chemical processing. For this research, the main purpose is to find the best approach to separate CH<sub>4</sub> and CO<sub>2</sub> in order to recover the high recovery and purity of CO<sub>2</sub>. Adsorption process was coined by a German physicist since 1881 (Kayser, 1881). Then this process had been further developed into different process and one of it was

PSA. Pressure swing adsorption is a technology using adsorption to recover gases based on the porosity of material used as adsorbent depending on the pressure and temperature of the feed and the selectivity of the adsorbent (Plants, 2016; Ebner and Ritter, 2004; Yang et al., 2001; Chai et al., 2011).

PSA is a periodic process. When the pressure of the system is reduced, it means the regeneration of adsorbent is occurring. The term 'swing' symbolizes the total pressure of the system changes from high pressure while feeding into system to low pressure while regeneration process (Ruthven et al., 1994; Tondeur and Wankat, 1985). The bed is pressurized with feed mixture to produce or separate desired product and alternatively, the other bed is undergoing regeneration process to remove the gas from the adsorbent in the bed (Grande, 2012).

### **2.3 Adsorbent**

Adsorbent is a substance that adsorb adsorbate on its surface. The major types of adsorbents in use are activated alumina (Ghorai and Pant, 2005; Lin and Wu, 2001), silica gel (Wang and LeVan, 2009), activated carbon (Namasivayam and Kavitha, 2002; Hameed et al., 2007), molecular sieve carbon (Kohlenstoff-Molekularsieb, 1970; Reid and Thomas, 1999), zeolites (Alver and Metin, 2012) and polymeric adsorbents (Lia et al., 2002; Zhang et al., 2003).

Kim et al. (2015) used carbon molecular sieve as adsorbent to study the separation of binary mixture of biogas that contained CH<sub>4</sub> and CO<sub>2</sub> via PSA process. The result showed a very high purity which is 97% of CH<sub>4</sub>. Other than that, zeolite is one of a common adsorbents too. Gholipour and Mofarahi (2016) used Zeolite to

research on the separation effects of binary mixture of natural gas with same ratio. The result for recovery of CO<sub>2</sub> is 95%.

There are 4 types of adsorbent were selected for this research which are palm kernel, activated charcoal, UiO-66 and raw Hydrotalcite.

### **2.3.1 Activated Charcoal (AC)**

Activated charcoal has porous surface that has a negative electric charge that causes positive charge gases to bond with it. Activated charcoal works by trapping toxins and chemicals in tiny pores which is also name adsorption. Activated charcoal was efficiently utilized as an adsorbent for the removal of hazardous dyes from the aqueous solutions (Iqbal and Ashiq, 2007; Mohan and Karthikeyan, 1997). The maximum surface is around 199 to 2105 m<sup>2</sup>/g BET surface area. Besides, activated charcoal was able to detoxify certain types of chemical via adsorption process (Canilhaa et al., 2004; Mussatto et al., 2004). Grande et al. (2013) utilized activated carbon as adsorbent to separate CH<sub>4</sub> and CO<sub>2</sub> under high pressure condition. The result showed higher selectivity towards CH<sub>4</sub> because the amount of CH<sub>4</sub> uptake was 7.86 mmol/g then CO<sub>2</sub> was 3.86 mmol/g.

### **2.3.2 Zirconium-benzenedicarboxylate (UiO-66)**

UiO-66 is sourced from zirconium salts and it is a metal organic framework (MOF) adsorbent (Cmarik et al., 2012; Chang and Yan, 2012). MOF offers a variety of topologies, porous networks and high surface areas which has high potential for the applications for adsorption or separation (Wang et al., 2015). It can be applied in gas



adsorption and separation (Yang et al., 2011; Wu et al., 2013; Lau et al., 2013). The maximum surface is around 1067 m<sup>2</sup>/g BET surface area and the volume of pore is 0.40 cm<sup>3</sup>/g. Cao et al. (2015) studied the CO<sub>2</sub> recovery by using UiO-66. The researcher fed in pure CO<sub>2</sub> and the end of result showed the CO<sub>2</sub> volume uptake is 91%.

## **2.4 Breakthrough Study on Adsorption of Carbon Dioxide (CO<sub>2</sub>)**

### **2.4.1 Effects of Gas Composition**

Shin (1995) studied the separation of binary gas mixture of natural gas with the ratio of half CH<sub>4</sub> and half CO<sub>2</sub>. His findings showed recovery and purity of CO<sub>2</sub> were very high which were about 96.79% and 99.12% respectively. The operating condition is at atmospheric pressure and adsorbent used were 5 Å Zeolite. Aquino et al. (2016) also used the similar gas composition and adsorbent with Shin which is 5:5, the different part was the operating pressure which was 400 kPa. However, the recovery of CO<sub>2</sub> was only 52%. Other than them, Mariem Kacema et al. (2015) used half CH<sub>4</sub> with half CO<sub>2</sub> and same adsorbent too to study the separation performance on natural gas mixture, the recovery obtained was 80% with a purity of near to 90%.

Yua et al. (2012) used another gas ratio which were 60% of CH<sub>4</sub> and 40% of CO<sub>2</sub>. Li et al. (2014) used MOF-5 while Yua et al. (2012) used carbon molecular sieve as adsorbents. Both of them operated breakthrough study at atmospheric pressure. Li obtained the CO<sub>2</sub> recovery of 98% while Yua et al. (2012) obtained around 30% of recovery varying with temperature.

Chou and Chen (2004) used the gas ratio of CH<sub>4</sub> to CO<sub>2</sub> 8:2 for his breakthrough study. The result was 90% at atmospheric pressure too. However, the adsorbent used was a common adsorbent which was activated carbon. Besides, Britt et al. (2009) used the same ratio with Mg-MOF-74 adsorbent but at relative lower pressure and the result obtained is 99.99% of CO<sub>2</sub> purity.

## **2.5 Carbon Dioxide (CO<sub>2</sub>) Adsorption through Pressure Swing Adsorption (PSA)**

### **2.5.1 Effects of Gas Composition**

Ko et al. (2005) utilized the PSA technique to study the separation of CO<sub>2</sub> from flue gas. The mixture used in experiment contained 15% of CO<sub>2</sub> and resulted very high recovery of CO<sub>2</sub> which is 97%. Zeolite was used for this study.

On the other hand, Pevida et al. (2016) selected 5:5 ratio on natural gas binary mixture by using activated carbon. Coal mine methane gas containing 45–55 vol% CH<sub>4</sub>, 7–8 vol% O<sub>2</sub>, 2–3 vol% CO<sub>2</sub> and balance nitrogen gas was studied by Olajossy et al. (2003) for separation purpose by the similar adsorbent with Pevida et al. (2016).

64% of CH<sub>4</sub> and 28% of CO<sub>2</sub> mixture were used by Asadi et al. (2013) and high purity of CO<sub>2</sub> can be produced after PSA process. Asadi et al. (2013) used MOF type of adsorbent for the research.

### **2.5.2 Effects of Initial Pressure**

Cavenati et al. (2006) studied PSA performance by using high pressure to remove CO<sub>2</sub> from natural gas. The pressure used was 500 kPa and the performance of the removal was good by using zeolite 14x. Uchida et al. (2001) also used relative high pressure of 300 kPa to study PSA performance of flue gas using zeolite. The recovery of CO<sub>2</sub> was 95% at the product. Labus et al. (2014) obtained a result of 68% as CO<sub>2</sub> recovery. The research was also conducted at 6 bar.

There were also some other researches focus on low pressure performance on PSA process. Webley and Zhang (2008) and Choi et al. (2003) both used a pressure closed to atmospheric pressure to study on the adsorption effect on PSA. Both of their result were moderate because the recovery was higher than 60%.

## CHAPTER THREE

### MATERIALS AND METHODS

This chapter writes up the materials needed and experimental procedures for the project. In order to understand the adsorption process and the adsorbent behaviour, characterization methods, instrument used and breakthrough analysis of the CO<sub>2</sub> are also described in this chapter.

#### 3.1 Research Methodology

The overall experimental activities carried out in this study are presented in Figure 3.1.

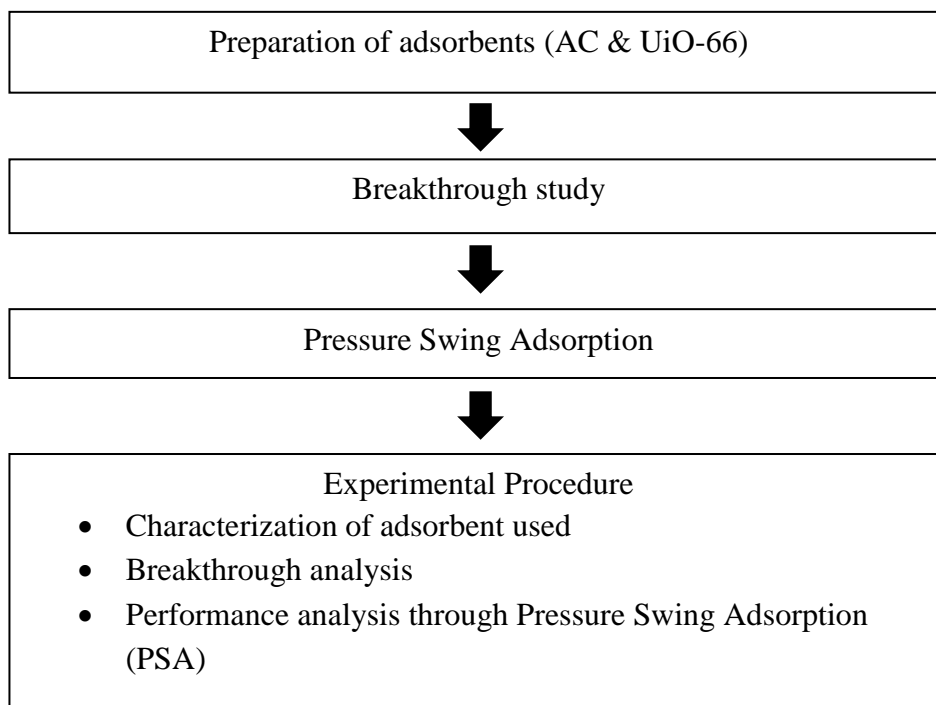


Figure 3.1: Process flow chart for research.

## 3.2 Chemicals

The main purpose of this research was to study the performance of CO<sub>2</sub> removal from natural gas by using different type of adsorbents. Hence, the main composition of natural gas were methane and carbon dioxide. These 2 gases were obtained from supplier with a very high purity (99.9%). During the experiment, the ratio of these 2 gases was controlled and mixed before entering adsorption column. The mixture of gases was allowed mixer to ensure perfect mixing before entering the column. The ratio of gases mixture used for CO<sub>2</sub>:CH<sub>4</sub> was varied.

### 3.2.1 Chemicals for Gas Mixture

Both of the tables below show the gases used for breakthrough study and PSA process. Those gases are CH<sub>4</sub> and CO<sub>2</sub> respectively which is also binary mixture of natural gas. Table 3.1 and Table 3.2 show the properties of CH<sub>4</sub> and CO<sub>2</sub>.

Table 3.1 Properties of Methane.

<b>Properties</b>	
Common name	Methane
Molecular formula	CH <sub>4</sub>
Molecular weight	16.04 g/mol
CAS number	74-82-8
Chemical structure	$\begin{array}{c} \text{H} \\   \\ \text{H}-\text{C}-\text{H} \\   \\ \text{H} \end{array}$

Table 3.2: Properties of Carbon Dioxide.

<b>Properties</b>	
Common name	Carbon Dioxide
Molecular formula	CO <sub>2</sub>
Molecular weight	44.01 g/mol
CAS number	120-38-9
Chemical structure	$\begin{array}{c} \ddot{\text{O}} \\ \parallel \\ \text{C} \\ \parallel \\ \ddot{\text{O}} \end{array}$

### 3.2.2 Chemicals for Adsorbent

In this research, 2 types of adsorbents were used which are activated charcoal and UiO-66. Tables below show all the chemicals used to prepare each adsorbents for this research use. Table 3.3 and 3.4 show the composition of adsorbents.

Table 3.3: The composition of Activated Charcoal.

Material	Chemical Formula	Supplier
Charcoal tablet	-	Biosis

Table 3.4: The composition of UiO-66.

Material	Chemical Formula	Supplier
Zirconium (IV) Chloride	ZrCl <sub>4</sub> , anhydrous powder, 99.99%	Sigma-Aldrich
Dimethylformamide (DMF)	HCON(CH <sub>3</sub> ) <sub>2</sub> , 99.8%	Sigma-Aldrich
1,4 benzenedicarboxylic acid (BDC)	C <sub>9</sub> H <sub>6</sub>	Sigma-Aldrich
Ethanol	CH <sub>3</sub> CH <sub>2</sub> OH, 99.8%	Sigma-Aldrich

### 3.3 Equipment

Different types of equipment were used for different purposes in this research such as preparation of adsorbents, breakthrough study and PSA process.

#### 3.3.1 Preparation of Adsorbents

During the preparation of adsorbents, a furnace was used to heat up the raw material. The raw material was then activated by flowing nitrogen gas into it and processed into adsorbent. The model number of furnace is GSL-1100X from MTI Corporation.



Figure 3.2: Furnace (GSL-1100X, MTI Corporation).

### 3.3.2 Breakthrough Study

The main equipment used for breakthrough study was a gas chromatography (GC). The purpose of gas chromatography was to observe the breakthrough time and breakthrough behaviour of the gases. The model of gas chromatography is 7890A from Agilent Technologies. Other than that, different kinds of valve were used to construct a mixer and column structure such as ball valve and globe valve.



Figure 3.3: Gas chromatography (7890A, Agilent Technologies).



### 3.3.3 Pressure Swing Adsorption (PSA) Process

The PSA structure mainly contained valves, flow meter and column bed. An example of simple schematic diagram for experimental setup for PSA is shown at below.

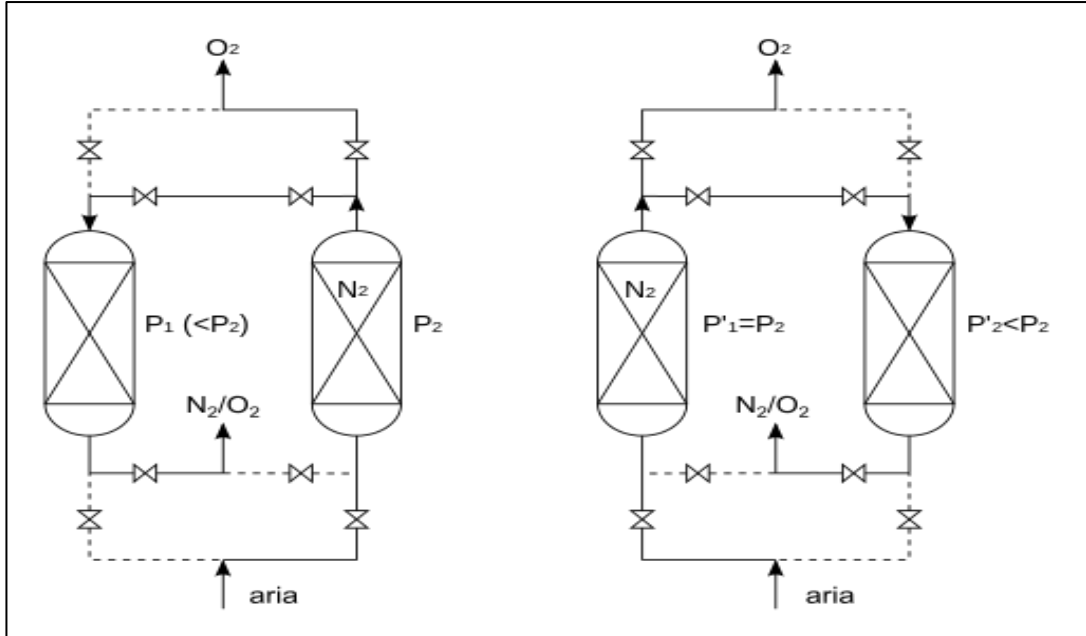


Figure 3.4: Schematic diagram of experimental setup for PSA (Corporation, 2016).

### **3.4 Characterization of Adsorbent**

The main purpose of characterization was to investigate the adsorbents' characteristics that affect the performance of adsorption by using different techniques. Characteristics of samples were analysed by 4 different tests which were Particle Size Analyzer, X-Ray Diffraction (XRD), Thermal Gravimetric Analysis (TGA), Brunauer-Emmett-Teller (BET) and Scanning Electron Microscopy (SEM) (Asuquo and Martin, 2016).

#### **3.4.1 X-Ray Diffraction (XRD)**

The model number of XRD is an effective analytical technique for phase identification of a crystalline material that can provide information of unit cell dimensions. This method is common for the study of crystal structure and atomic spacing. X-ray diffraction is based on constructive interference of monochromatic X-ray and the crystal sample. The model of equipment is D8 from Bruker. Hence, the interaction between X-ray and crystal sample produces constructive interference to study about crystal structure.

### 3.4.2 Thermal Gravimetric Analysis (TGA)

TGA is a method for thermal analysis in which studies the changes in chemical and physical properties of the materials with the increase in temperature as a function of time. It is a common method to investigate mass loss or gain due to decomposition, oxidation, or gain due to decomposition, oxidation or loss of moisture. In summary, it is a characterization method through analysis of characteristic decomposition patterns of the specific material. The figure below shows the model of equipment which is TGA 7 from Perkin Elmer.



Figure 3.5: Thermogravimetric Analyzer (TGA 7, Perkin Elmer).

### 3.4.3 Brunauer-Emmett-Teller (BET)

In order to study the pore size distribution, BET is a suitable method to find out the specific surface area of a sample. This technique aims to study the physical adsorption of gas molecules on a solid surface and hence it is an important analysis for the measurement of the specific surface area of a material. The model of BET analyser is ASAP 202 from iDB. It applies the Langmuir theory which explains the monolayer adsorption.



Figure 3.6: Surface and porosity analyzer (ASAP 2020, iDB).

#### **3.4.4 Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX)**

The working principle of SEM uses electron emission for imaging the condition of surface of material. SEM uses a beam of electrons to produce a variety of signals towards the surface of material. It is like a light microscope uses visible light. This characterization method can produce high-resolution imaging of surface especially the porosity of the material's surface. In this project, the magnification used is x100 times in order to get the suitable image that shows the porosity for study.