



## CHAPTER III EXPERIMENTAL

### 3.1 Materials

Ultem (polyetherimide) was donated by UOP LLC., USA. Activated carbon purchased from Aldrich was put into mixed matrix membranes as molecular sieve particles. Acetone and N-methylpyrrolidone (NMP) purchased from Labscan Co., Ltd. were used as solvents. Polyethylene glycol (PEG), 1,2-, 1,3-, 1,4- and 2,3-butanediol purchased from Aldrich were selected as liquid additives.

### 3.2 Methodology

#### 3.2.1 Preparation of Liquid Additive Adsorbed Activated Carbon

A quantity of each liquid additive used for impregnation into pores of activated carbon was studied by varying it from 10 to 40 wt%. The next step was to evaporate an excess liquid additive from the activated carbon at 150°C in an oven for 24 hr. The results were reported as a phase of each liquid adsorbed activated carbon.

#### 3.2.2 Preparation of Dense Membrane

All membranes were prepared by the solution-casting method. Ultem solution was prepared by admixing Ultem with NMP solvent and stirring for 12 hr. The membrane solution was degassed by a vacuum pump. Then bubble free solution was casted onto a clean glass plate and adjusted the thickness of membrane by a casting-knife. The resultant membrane was allowed to slowly evaporate for overnight until a film was formed on the glass plate surface. The film was dried and annealed at 80°C for 12 hr in an oven in order to remove NMP solvent out of the thin film sheet. The dried membrane was put between two glass plates to prevent curling of the membrane film, and left it to form a stable membrane for 48 hr.

#### 3.2.3 Preparation of Dense Mixed Matrix Membranes

##### 3.2.3.1 *Solid Mixed Matrix Membrane*

All experiment steps were followed the preparation of Ultem membrane except that activated carbon was added into NMP solvent before Ultem in order to get rid of air inside the pores of activated carbon.

#### *3.2.3.2 Solid-Liquid Mixed Matrix Membrane*

Each type of liquid additives and acetone were firstly added into activated carbon, NMP solvent and Ultem were added afterward, and then followed by the preparation steps of Ultem membrane.

#### 3.2.4 Preparation of Asymmetric Membrane

All membranes were prepared by the solution-casting method. Ultem solution was prepared by admixing Ultem with NMP solvent and stirring for 12 hr. The membrane solution was degassed by a vacuum pump. Then a bubble free solution was casted onto a clean glass plate and adjusted the thickness of membrane by a casting-knife. The resultant membrane was allowed to evaporate at room temperature. After an additional set time of 30 sec, the detached membrane was gelled in an iced water bath at 3-4°C for 1 hr and subsequently annealed in a hot water bath at 80-90°C for 1 hr to remove any residual NMP. Finally, the membrane was dried at 90°C for 24 hr. Paper towels were placed between glass plates and the membrane film, and left to form a stable membrane for 48 hr.

#### 3.2.5 Preparation of Asymmetric Mixed Matrix Membranes

##### *3.2.5.1 Solid Mixed Matrix Membrane*

All experiment steps were followed the preparation of Asymmetric Membrane except that activated carbon was added into NMP solvent before Ultem in order to get rid of air inside the pores of activated carbon.

##### *3.2.5.2 Solid-Liquid Mixed Matrix Membrane*

Each type of liquid additives and acetone were firstly added into activated carbon, NMP solvent and Ultem were added afterward, and then followed by the preparation steps of Asymmetric membrane.

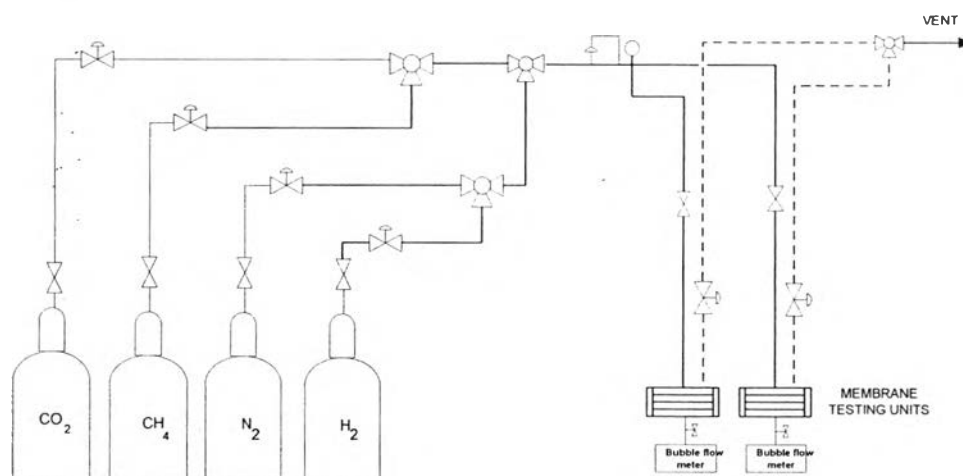
#### 3.2.6 Gas Permeability Measurements

In this experiment, CO<sub>2</sub> (Prax Air), N<sub>2</sub> (Prax Air), H<sub>2</sub> (Prax Air) and

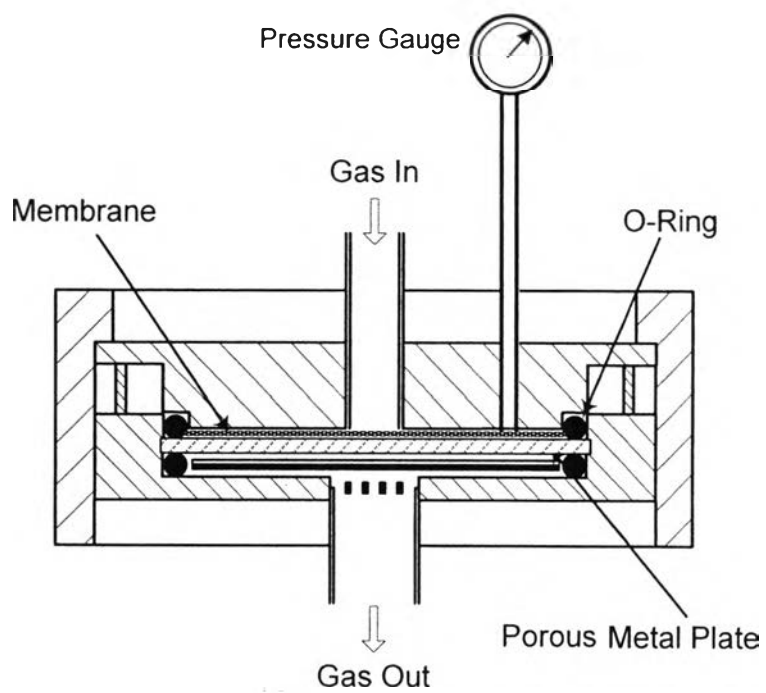
CH<sub>4</sub> (TIG) were tested with all types of membranes. All tested gases were in a high purity (HP) grade and used as received.

The schematic diagram of gas permeability measurement is shown in Figure 3.1. The experimental setup included gas sources, membrane testing units and a bubble flow meter. Schematic of the membrane testing unit is shown in Figure 3.2.

Single gas permeability measurement of H<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub> and CO<sub>2</sub> were determined at 25°C in sequence by using the membrane testing unit in which the membrane was placed on a porous metal plate and pressurized to the desired pressure at the feed size. The pressure difference across the membrane was maintained at 100 psi. It was due to a very low permeability of gases that pass through the membrane. Once reached the steady-state, an individual gas flow rate was measured using a soap bubble flow meter. The certain data were used to calculate permeability and selectivity of gases.



**Figure 3.1** Schematic of the experimental setup for determining a gas permeability.



**Figure 3.2** Schematic of the membrane testing unit.