

## CHAPTER III EXPERIMENTAL

### 3.1 Materials

Cellulose acetate (Aldrich) with a number-average molecular weight of 30,000 and 39.8% acetyl content was selected as the continuous phase. Acetone purchased from Labscan Co., Ltd. was used as solvent. All reagent-graded chemicals were used as received. The following zeolites, namely, NaX, NaY, NaA, AgA, CaA silicalite and beta were kindly provided by UOP LLC., USA. All zeolites were calcined at 300°C for 3 h to remove any impurities before use. The selected physical properties of zeolites used in this work are shown in Table 3.1.

**Table 3.1** Selected physical properties of zeolites used in this work (Huang *et al.*, 2006, Li *et al.*, 2007, Lonyi and Valyon, 2001, and Wang *et al.*, 2001).

Zeolite type	Si/Al ratio	Pore size (Å)
NaY	2.5	7.4
NaX	1.3	7.4
Silicalite	196	5.2×5.7
Beta	16	7.1×7.3
NaA	1.0	3.8
AgA	1.0	~3.8
CaA	1.0	4.5
H-mordenite	6.8	6.7×7.0

### 3.2 Methodology

#### 3.2.1 Preparation of Mixed Matrix Membrane

Membranes were prepared by solution-casting method. Each type of zeolites was dissolved in acetone and stirred for 3 h. After that, the zeolite mixed solution was ultrasonicated for 10 min to improve the dispersion of zeolite particles in the so-

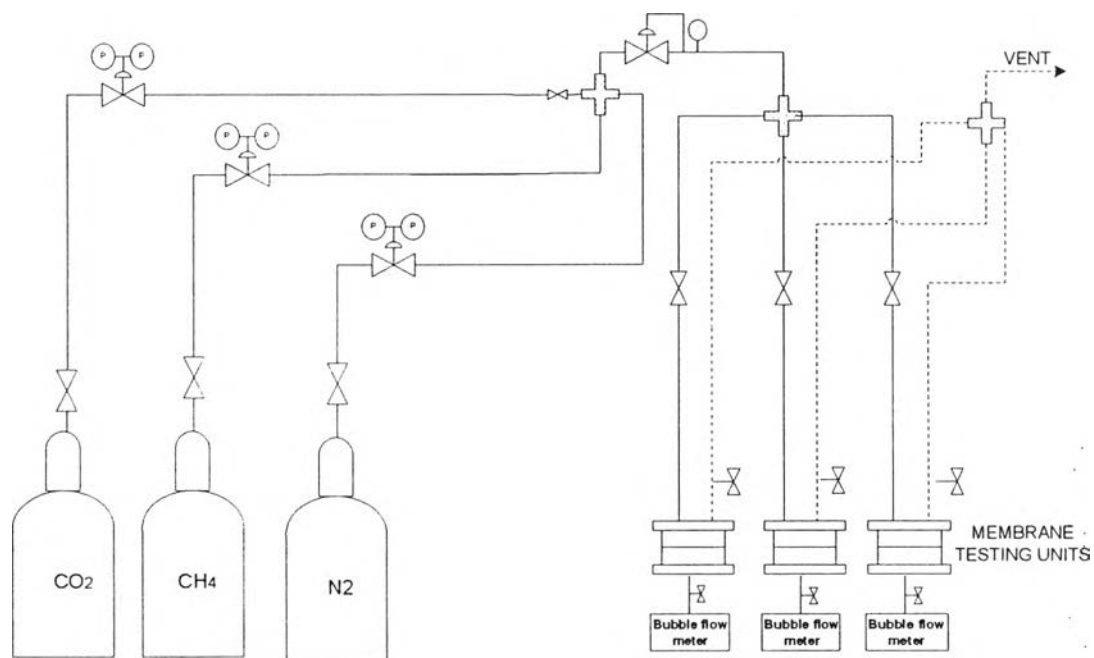
lution. Zeolite particles were then “primed” by adding approximately 15 wt% of total amount of CA, which was suggested to increase the compatibility between zeolite and polymer, and to minimize the aggregation of zeolite particles. The mixture was stirred overnight on a magnetic stirrer to enhance the homogeneity. After the remaining CA was added, final mixture was further mixed for 2 h. The concentration of CA in acetone was 15 % (w/v). The concentrations of each zeolite in CA were varied with an interval of 10% from 0% (w/w) to 40% (w/w) on solvent-free basis. The casting solution was cast on a clean glass plate at room temperature using a casting blade [2]. After an additional set time of 20 sec, the detached membrane was gelled in an iced water bath at 3-4°C for 1.5 h and subsequently annealed in a hot water bath at 65-75°C for 1 h to remove any residual acetone. Finally, the membrane was naturally dried at room temperature for 72 h in between paper towels and two glass plates to prevent curling of the membrane.

### 3.2.2 Gas Permeability Measurements

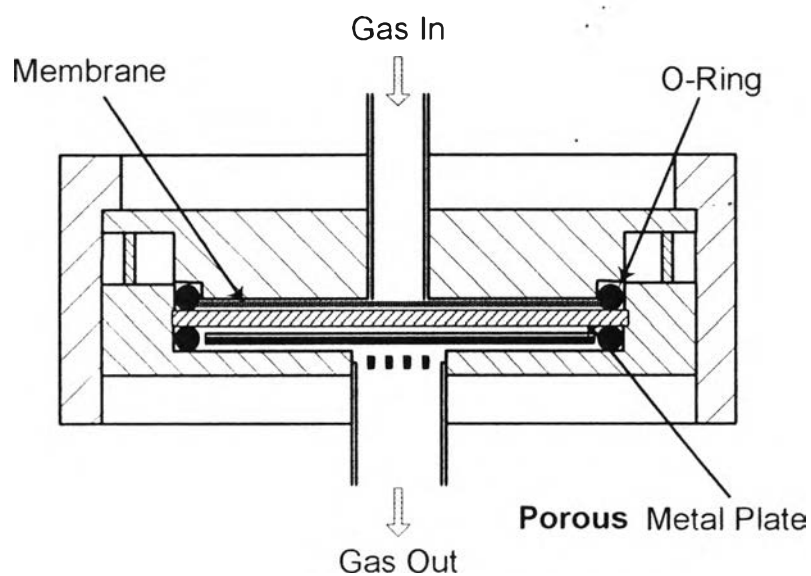
In this study, CO<sub>2</sub> (Prax Air) and CH<sub>4</sub> (TIG) were used as tested gases for all membranes; however, for NaA-CA MMMs, O<sub>2</sub> and N<sub>2</sub> (TIG) were additionally measured. All tested gases were in a high purity (HP) grade and used as received.

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

Single gas permeabilities of O<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 a gas permeation 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 50 and 100 psi. Once reached the steady-state, individual gas flow rates were measured using a soap bubble flow meter. The attained data were used to calculate the gas permeance and selectivity.



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



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