



CHAPTER III EXPERIMENTAL

3.1 Materials and Chemicals

1. Fittings and valves
2. Vacuum pump
3. Data logger
4. Carbon dioxide gas (purity 99.99% purchased from Praxair Inc.)
5. Activated carbon (supported by Carbokarn Co., Ltd)
6. Ethylene imine polymer solution (PEI) 50 wt% in water (purchased from Sigma-Aldrich) with a high molecular weight ($M_w = 600,000-1,000,000$)
7. Methanol (purity 99.95 % purchased from Praxair Inc.)

3.2 Experimental Procedures

3.2.1 Impregnation

- a. AC was ground and sieved to obtain a particle size of 20-40 mesh.
- b. AC was dried at 120 °C for 6 hrs for removing moisture.
- c. AC was added to PEI solution with methanol of desired PEI initial concentration (0.1 – 2.0 g/L). The solid to liquid ratio was 1 g of AC to 20 mL of PEI solutions in a closed system.
- d. The AC together with the PEI solutions were agitated in an orbital shaker at 180 rpm at 25 °C for 3 days.
- e. The adsorbents were dried at 120 °C for 3 hrs for removing volatile solvent and moisture.

3.2.2 Characteristics of Adsorbent

- a. Surface areas and pore volumes of the adsorbents were measured with the BET method on a Quantachrom/Autosorb1-MP instrument. Each adsorbent was first out gassed to remove the humidity on its surface under vacuum at 25 °C for 12 hrs prior to the analysis.

After that, N₂ was purged to adsorb on its surface. The volume-pressure data was used to calculate the BET surface area.

- b. Perkin-Elmer/Pyris Diamond TG-DTA instrument was used to study thermal decomposition of adsorbents in order to evaluate the actual amount of loading. Each adsorbent was heated to 600 °C with a ramping rate of 10 °C/min in N₂ atmosphere (100 mL/min flow-rate).
- c. The surface organic spectra were measured with the Thermo Nicolet/Nexus 670 FTIR instrument. The sample was dried in the oven at 120 °C prior to be mixed with KBr powder. The sample was run in the ratio mode allowing for subtraction of a pure KBr baseline. The sample chamber was purged with nitrogen during the entire experiment. The spectrometer collected 64 spectra in the range of 400–4000 cm⁻¹ with a resolution of 4 cm⁻¹.
- d. Shimadzu/UV-1800 UV-Vis Spectrophotometer was used to investigate the PEI concentration.

3.2.3 Adsorption Measurement

The schematic diagram of the experimental set-up is shown in Figure 3.1. A pressure transmitter was installed to measure pressure of the system. One gram of the prepared adsorbent was loaded into the stainless steel adsorption chamber, which was heated by the furnace in order to reach the adsorption temperatures. He (Ultra high purity, Praxair Inc.) was used as a purge gas in this study. The adsorption processes were carried out using high purity CO₂ gas (99.99%). Effects of adsorption temperature were investigated by varying the temperature from 30 to 75 °C within a pressure range of 0–1 atm. The temperature of the adsorption chamber was adjusted and maintained by an internal temperature sensor.

3.2.4 Desorption Measurement

CO₂ desorption measurements were performed after the adsorption. The adsorbent, approximately 1 g, was packed into the reactor in a temperature

controlled furnace. The desorption experiments were then carried out by taking the adsorbent out of the reactor for heating at 120 °C for 1 hrs to remove adsorbed CO₂ and volatile components (Goeppert *et al.*, 2011; Ma *et al.*, 2009). These adsorption/desorption cycles were repeated three times.

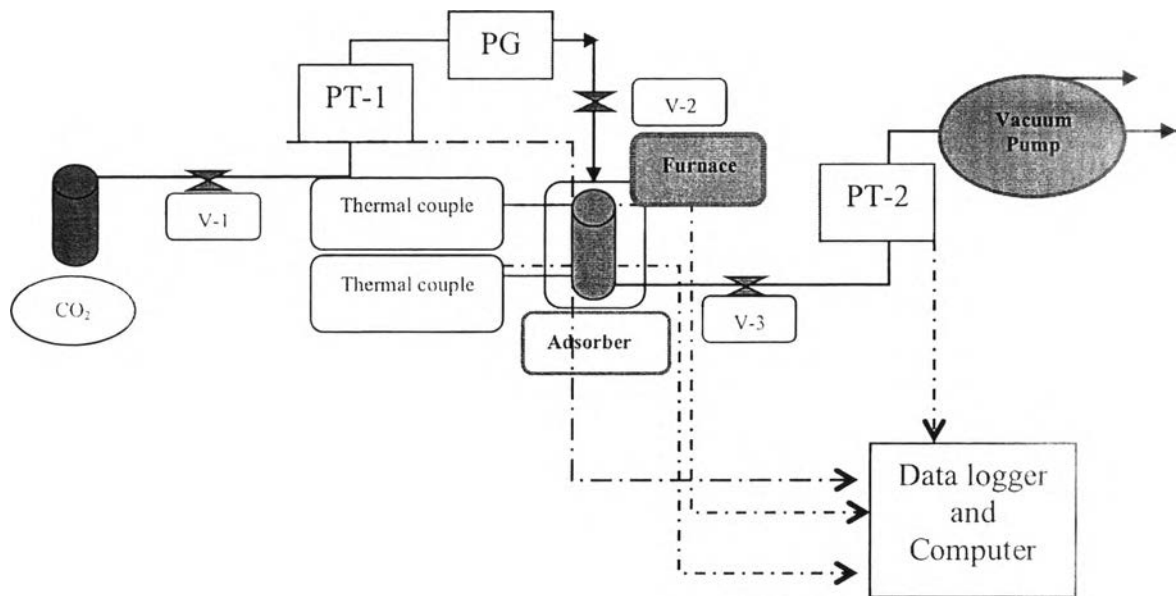


Figure 3.1 Schematic of experimental set-up for the adsorption of CO₂.

The amount of carbon dioxide adsorption was determined by Eq. (3.1)

$$n_i = \frac{P_1(V_1+V_2)}{ZRT} - \frac{P_2(V_1+V_2)}{ZRT} \quad (3.1)$$

where,

n_i = mole of adsorbed CO₂, mol

P_1 = pressure of the system before equilibrium, atm

P_2 = pressure of the system after equilibrium, atm

V_1 = volume of a manifold, cm³

V_2 = volume of a cylinder with adsorbent, cm³

Z = compressibility factor

R = 82.05 cm³ atm/mol K

T = temperature of the sample, K