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 carbons (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. PEI (M_w = 2,000) solution 50 wt% in water (purchased from Sigma-Aldrich)
8. Liquid PEI (M_w= 25,000) (purchased from Sigma-Aldrich)
9. Methanol (purity 99.95 % purchased from RCI Labscan Limited)
10. Helium gas (purity 99.999% purchased from Praxair Inc.)

3.2 Experimental Procedures

3.2.1 Adsorbent Impregnation

a. ACs were ground and sieved to obtain a particle size of 20-40 mesh.

b. ACs were dried at 120 °C for 24 hrs for removing moisture.

c. ACs were added to PEI solution or liquid PEI (with various M_w) with methanol of desired PEI initial concentration (1.0 – 5.0 g/L). The solid to liquid ratio was 1 g of ACs to 20 mL of PEI solution in a closed system.

d. The ACs together with the PEI solution were agitated in an orbital shaker at 180 rpm at 25 °C for 3 days.

e. The adsorbents were dried at 120 °C for 24 hrs for removing volatile solvent and moisture.

3.2.2 Adsorbent Characterization

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 80 °C for 12 hrs prior to the analysis. After that, N_2 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 1000 °C with a ramping rate of 10 °C/min in N_2atmosphere (100 mL/min flow-rate).

c. The surface organic spectra was 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 was collect 64 spectra in the range of 800–3200 cm^{-1} with a resolution of 4 cm^{-1}.

d. Shimuszu/UV-1800 UV-Vis Spectrophotometer was used to determine the left PEI solution concentration.

3.2.3 Adsorption Measurement

The schematic diagram of the experimental set-up was 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 CO_2 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 Regeneration

After adsorption, the regeneration of spent adsorbent was carried out by taking the adsorbent out of the reactor for heating at 120 °C for 3 hrs to remove adsorbed CO_2 and volatile components (Ma *et al.*, 2009; Goeppert*et al.*, 2011). These adsorption/desorption cycles were repeated three times.

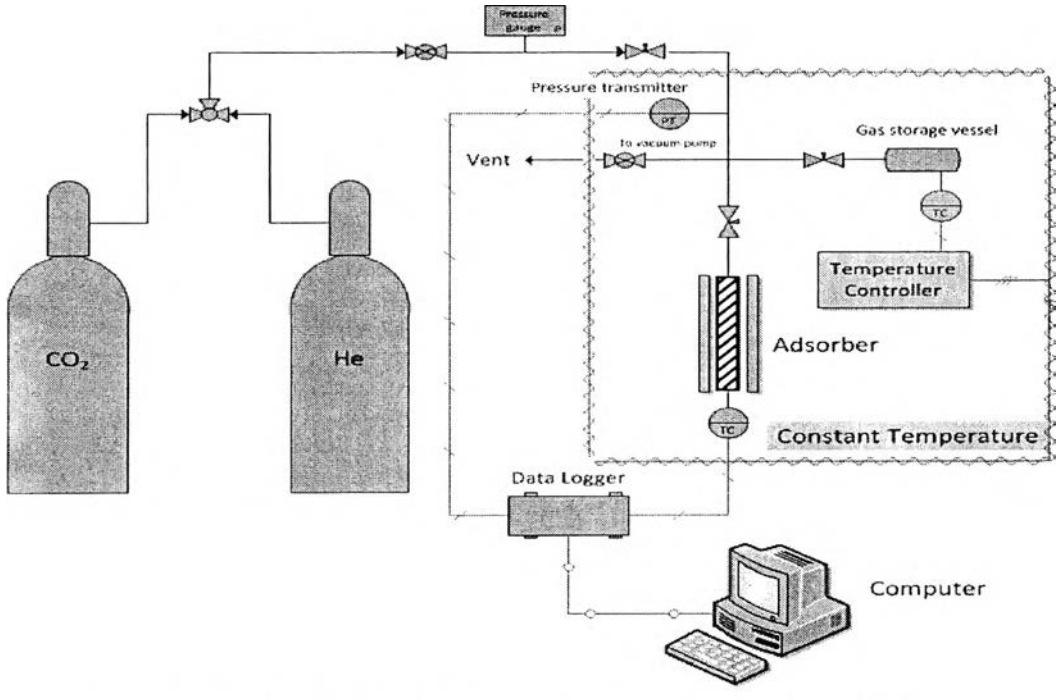

Figure 3.1 Schematic of experimental set-up for the adsorption of CO_2.

The amount of CO_2 adsorption was determined by

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

where,

n_i= mole of adsorbed CO_2, 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, cm^3

V_2= volume of a cylinder with adsorbent, cm^3

Z = compressibility factor

R = 82.05 cm^3atm/mol K

T = temperature of the sample, K