

CHAPTER III EXPERIMENTAL

3.1 Materials and Chemicals

1. Fittings and valves
2. Vacuum pump
3. Data logger
4. Carbon dioxide gas, CO₂ (purity 99.99% purchased from Praxair Inc.)
5. Activated carbon, AC (supported by Carbokarn Co., Ltd.)
6. Paraformaldehyde (prilled, 95% purchased from Aldrich Co., LLC)
7. Phenol (99% purchased from Aldrich Co., LLC)
8. Tetraethylenepentamine, TEPA (purchased from Aldrich Co., LLC)
9. Methanol (purity 99.95 % purchased from RCI Labscan Limited)
10. Chloroform, (AR grade purchased from RCI Labscan Limited)
11. Sodium hydroxide, NaOH (1.0 N purchased from Fluka)
12. Helium gas. He (purity 99.999% purchased from Praxair Inc.)

3.2 Experimental Procedures

3.2.1 Synthesis Benzoxazine Monomer

- a. TEPA, phenol, and paraformaldehyde at a 1:2:4 molar ratio were mixed together in an aluminium cup at 100 °C.
- b. The mixing continued at 100 °C for 60 min until it became transparent pale yellow.
- c. The synthesized product was washed with NaOH aqueous solution and water.
- d. The synthesized product was dried under vacuum at 50 °C and 25 mbar.

3.2.2 Grafting Adsorbent

- a. AC was ground and sieved to obtain a particle size of 20-40 mesh.

b. AC was dried at 120 °C for 24 h for removing moisture.

c. Benzoxazine monomer was dissolved in methanol and chloroform.

After the addition of AC, the mixture was reacted at 180 °C for 1 hr. AC was then collected with filtration, and dried under vacuum to give the product of polybenzoxazine grafted-ACs.

3.2.3 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 h 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 1000 °C with a ramping rate of 10 °C/min in N₂ atmosphere (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 will be collect 64 spectra in the range of 800–3200 cm⁻¹ with a resolution of 4 cm⁻¹. Moreover, It was use for confirm the chemical structure of benzoxazine monomer.

d. Differential scanning calorimetry (DSC) was used to determine the cure temperature of benzoxazine.

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

f. Scanning electron microscopy (SEM) was used to determine the morphology of adsorbent. The morphology of AC and PBZ grafted CSAC were assessed with the application of a scanning electron microscope (SEM; JEOL JSM-5410LV). The SEM was operated at 15.0 kV of an accelerating voltage. The samples were sputter-coated with gold prior to analysis.

3.2.4 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.

The amount of CO₂ adsorption was determined by

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

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 cm³atm/mol K

T = temperature of the sample, K

3.2.5 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 h to remove adsorbed CO₂ and volatile components (Ma *et al.*, 2009; Goepfert *et al.*, 2011). These adsorption/desorption cycles were repeated three times.

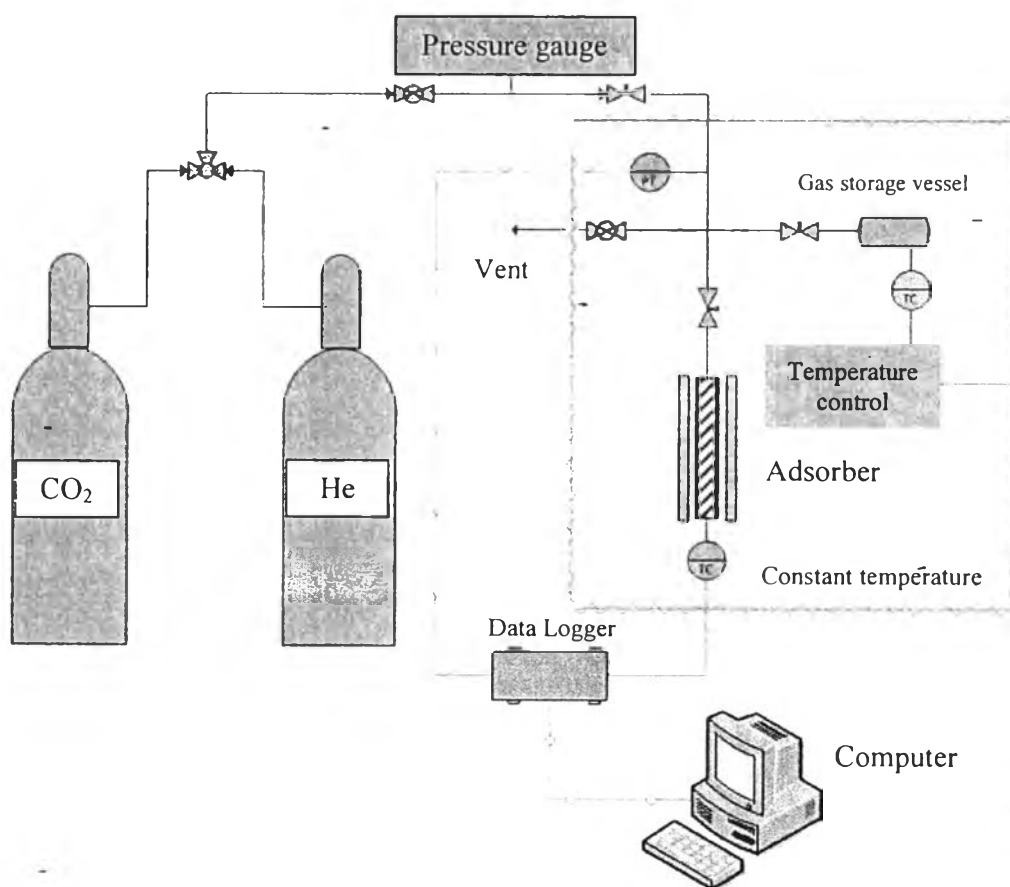


Figure 3.1 Schematic of experimental set-up for the adsorption of CO₂.