

CHAPTER III

EXPERIMENTAL

3.1 Materials and Chemicals

1. Fittings and valves
2. Mass flow controller
3. Temperature controller
4. Gas chromatograph
5. Ultrasonic bath
6. Coconut shell activated carbon (CSAC) (supported by Carbokarn Co., Ltd.)
7. Palm shell activated carbon (PSAC) (supported by Carbokarn Co., Ltd.)
8. Methane gas (99.99% purity purchased from Labgaz Thailand Co., Ltd.)
9. Carbon dioxide gas (99.99% purity purchased from Labgaz Thailand Co., Ltd.)
10. Helium gas (99.99% purity purchased from Praxair Inc.)
11. Sulfuric acid (98% purity purchased from RCI Labscan Ltd.)
12. Potassium hydroxide (85% purity purchased from RCI Labscan Ltd.)

3.2 Experimental Procedures

3.2.1 Adsorbent Preparation

- a. CSAC was grinded and sieved to obtain a particle size of 20-40 mesh.
- b. CSAC was dried at 120 °C for 24 hrs to remove moisture.

3.2.2 Adsorbent Characterization

- a. The surface area and total pore volume of the adsorbent was measured with the BET method on a Quantachrom/Autosorb1-MP instrument. The 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, nitrogen was purged to adsorb on its surface. The volume-pressure data was used to calculate the BET surface area.

- b. The morphology of the adsorbent was investigated by using the SEM, Hitachi S-4800, with an accelerating voltage of 2 kV and varying magnifications of 250, 1,000, 1,500, and 10,000. The adsorbent was coated with platinum under vacuum condition before observation.

3.2.3 Adsorption Measurement

The schematic diagram of the experimental set-up was shown in Figure 3.1. The adsorption kinetics of methane and carbon dioxide were carried out in a stainless steel packed bed column with an inside diameter of 7.0 mm at atmospheric pressure and room temperature. In the middle of the column, approximately 5.0 g of activated carbon was packed between glass wool. The volumetric flowrates of methane and carbon dioxide were controlled and monitored by a mass flow controller. Carbon dioxide composition was also varied from 10 to 30 vol%. The outlet gases (CH_4 and CO_2) from the column were analyzed both qualitatively and quantitatively by a Hewlett Packard 5890 series II gas chromatograph equipped with a thermal conductivity detector (TCD) using two packed columns, which were Hayesap D 100/120 and Molecular Sieve 5A 80/100. The sampling loop was at every 2.5 min. The oven and detector temperature of the gas chromatograph were 100 and 150 °C, respectively. Helium was used as a carrier gas and a diluting agent for the system. Pressure of the carrier gas, helium, was 70 psi.

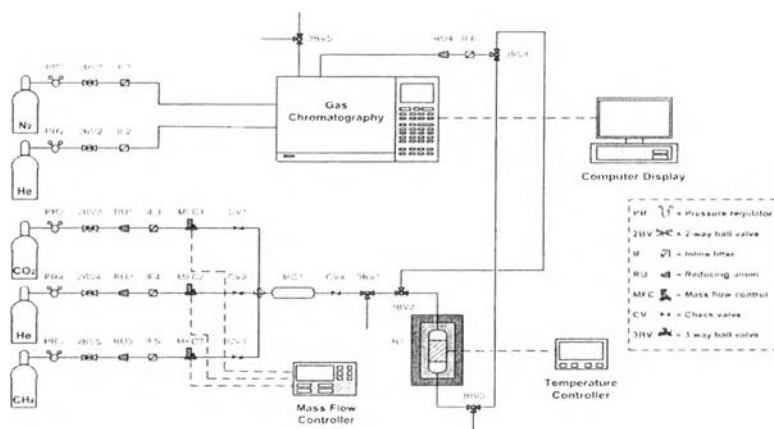


Figure 3.1 Schematic diagram of the experimental set up.

3.2.4 Adsorbent Stability

The adsorbent stability was studied by the adsorption-desorption of methane and carbon dioxide on the investigated adsorbents at atmospheric pressure and room temperature. The adsorption was first operated at the total flowrate of gas mixtures about 25 ml/min for 1 hr before the desorption, which was carried out at the helium flowrate of 50 ml/min for 1 h. After that, the same procedure was repeated three times for the adsorption-desorption process to study the adsorbent stability. The outlet gases from the column were analyzed both qualitatively and quantitatively by a Hewlett Packard 5890 series II gas chromatograph equipped with a thermal conductivity detector (TCD).

3.2.5 Adsorbent Surface Treatment

CSAC was grinded and sieved to obtain a particle size of 20-40 mesh (Sun *et al.*, 1996). The adsorbent surface was modified by acid, 98% sulfuric acid, and alkali, 85% potassium hydroxide, treatment. Approximately, 25.0 g of CSAC was sonicated in each 250 ml of 1.0 M sulfuric acid and potassium hydroxide solution for 45 min at 30 °C. Then, the prepared samples were washed adequately with deionized water until pH of filtered water was 7 and dried at 120 °C for 24 hrs to remove moisture.