

CHAPTER III EXPERIMENTAL

3.1 Materials and Equipment

3.1.1 Materials

- a. Fittings and valves
- b. Mass flow controller
- c. Temperature controller
- d. Coconut shell activated carbon (CSAC) (supported by Carbokarn Co., Ltd., Thailand)
- e. Palm shell activated carbon (PSAC) (supported by Carbokarn Co., Ltd., Thailand)
- f. Methane gas (99.99% purity purchased from Labgaz Thailand Co., Ltd.)
- g. Carbon dioxide gas (99.99% purity purchased from Labgaz Thailand Co., Ltd.)
- h. Helium gas (99.99% purity purchased from Praxair Inc.)

3.1.2 Chemicals

- a. Ammonia (25% purity purchased from RCI Labscan Ltd.)
- b. Sodium hydroxide (99% purity purchased from RCI Labscan Ltd.)
- c. Potassium hydroxide (85% purity purchased from RCI Labscan Ltd.)
- d. Nitric acid (70% purity purchased from RCI Labscan Ltd.)
- e. Sulphuric acid (98% purity purchased from RCI Labscan Ltd.)
- f. Phosphoric acid (85% purity purchased from RCI Labscan Ltd.)
- g. Methyl ester sulphonate (support by PTT Public Company Ltd.)

3.1.3 Equipments

- a. Gas chromatograph, Hewlett Packard 5890 series II
- b. Surface area analyzer (SAA), Quantachrom/Autosorb1-MP
- c. Scanning electron microscope (SEM), Hitachi S-4800

- d. Fourier transform infrared spectrophotometer
- e. Incubator shaker: Model SK-737, Amerex Instruments, Inc., U.S.A.
- f. Total organic carbon analyzer (TOC-V_{CSH}), Shimadzu

3.2 Experimental Procedures

3.2.1 Adsorbent Preparation

- a. CSAC was ground and sieved to obtain a particle size of 20-40 mesh.
- b. CSAC was dried at 120 °C for 24 h 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 h 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 15 kV and using magnification of 15,000. The adsorbent was coated with platinum under vacuum condition before observation.
- c. Zeta potential measurements of activated carbon were carried out with a Zeta Potential Analyzer (Zeta sizer 2000, Malvern). Approximately, 1 mg of powder samples was ground in an agate mortar, mixed with 10 ml distilled water, and settled for several minutes. The suspension with colloidal sized particles was collected to determine zeta potential after pH adjustment with 0.01M solutions of NaOH or HCl. The pH_{pzc} is the corresponding pH value when the zeta potential was 0 mV.
- d. Fourier transform infrared spectroscopy (FTIR) was used to qualitatively evaluate the chemical structure of carbon materials. The IR spectra were collected using a Nicolet, Nexus 670 FTIR

spectrometer. The activated carbon samples were grounded into fine powder and mixed with KBr. The mixture was used for the preparation of KBr pellets. The IR spectrum was obtained over a frequency between 400 and 4000 cm^{-1} .

- e. The TOC-V_{CSH}, Shimadzu, was used to investigate the concentration of MES for the MES adsorption on the activated carbon. In the adsorption measurements, MES solutions at different concentrations (from 152.8 ppm to 6.0 ppm) and pH 3 were used. The pH solution was adjusted by HCl and NaOH. 0.1 g of activated carbon was added to every 100 ml of surfactant solution prepared at different concentrations. The solution stirred at constant speed. At 60, 120, 180, and 240 min, the concentration of MES in solution were measured by the TOC.

3.2.3 Adsorption Measurement

The schematic diagram of the experimental set-up is 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.

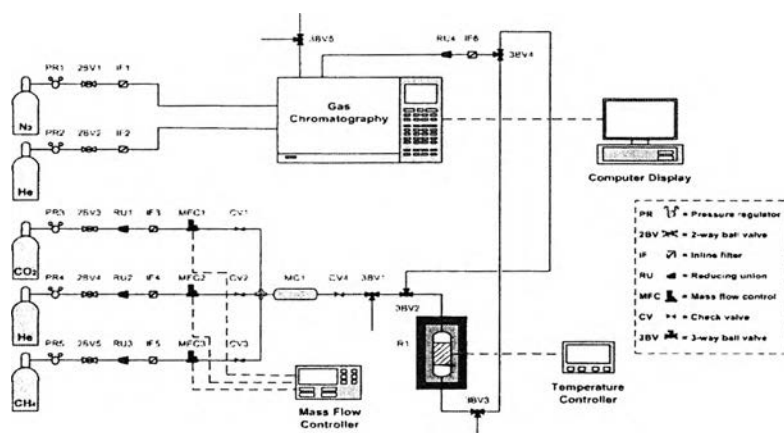


Figure 3.1 Schematic diagram of the experimental set up.

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 h 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 ground and sieved to obtain a particle size of 20-40 mesh. Before chemical treatment, the CSAC was purified by boiling in a water bath for 2 h and washed with deionized water to remove fine particles, and subsequently dried in an oven at 120 °C for 24 h. The acidic and alkaline solutions for preparation of CSACs were made with 10M HNO₃, 9M H₂SO₄, 7.3M H₃PO₄, 10M NaOH, 10M KOH, and 6.6M NH₃·H₂O. The CSAC was soaked in each solution at 70 °C for 2 h and were then placed on an incubator shaker at 35 °C for 24 h. The modified CSAC

was separated from the solution and washed repeatedly with distilled water. It was then dried in an oven at 120 °C for 24 h and transferred to a desiccators before use.

For MES modified CSAC, the AC was ground and sieved to obtain a particle size of 20-40 mesh. The CSAC was dried in an oven at 120 °C for 24 h. MES adsorption on the CSAC was first carried out. In the experiment, MES solutions at different concentrations (from 152.8 ppm to 6 ppm) and pH 3 were used. The pH solution was adjusted by HCl and NaOH. 5 g of activated carbon was added to every 5.0 L of surfactant solution prepared at different concentrations. The equilibrium adsorption of MES was reached in 180 min. The solution stirred at constant speed. After 180 min, the activated carbon in solution was filtered by filter paper and dried in vacuum oven at 70 °C for 12 h to remove excess water.