CHAPTER III
EXPERIMANTAL

3.1 Materials and Chemicals

1. Fitting and valve

2. Activated carbon (supported by Carbonkarn Co., Ltd.)

3. Ethylenediamine branched (average M_w ~800, average M_n ~600) (purchased from Aldrich)

4. Nitric acid 70% (density = 1.41, Mw = 63.01) (purchased from RCI Labscan)

5. Carbon dioxide gas (purity 99.99% purchased from Praxair Inc.)

6. Nitrogen gas (purity 99.99% purchased from Praxair Inc.)

7. Methanol (purchased from Macron Fine Chemicals^{TM})

3.2 Experiment Procedures

3.2.1 Surface Treatment

3.2.1.1 Oxidative Treatment

a. AC was ground and sieved to obtain a particle size of 70-140 mesh.

b. AC was dried at 110 °C overnight to remove moisture.

c. 1 g of AC was placed in a round bottom flask with 10 cm^3 of 5, 10 and 15 M nitric acid.

d. The content was stirred at 60 °C for different periods of time: 1, 4 and 8 h.

e. The samples were washed with purified water until the washing was no longer acid.

f. Finally, the samples were dried overnight in an oven at 110 °C.

3.2.1.2 Wet Impregnation

      a. The desire amount of PEI was dissolved in 4 g of methanol.

      b. The solution was mixed mechanically by several apparatus including shaker, stirrer and sonic bath for 15 min.

      c. 0.5 g of AC or OX samples were added into the PEI solution.

      d. The slurry was mechanically mixed for a further 30 min using the same apparatus specified in 3.2.2.2.b.

      f. The resulting slurry was dried overnight in an oven at 70 °C.

3.2.2 Characteristic and Analysis of the Adsorbent

      a. Netzsch Simultaneous Thermal Analyzer (STA) F3 Jupiter instrument was used to study thermal decomposition of adsorbents. Each adsorbent was heated to 800 °C with a ramping rate of 10 °C/min under 100 mL/min of N_2 flow.

      b. Sorptomatic surface area analyzer was used to measure surface area, pore volume and pore size of adsorbents. Surface area and pore volume were determined by Brunauer Emmett Teller (BET) method. Horvath Kawazoe (HK) method was applied to determine the micropore distribution. Prior to the analysis, the adsorbent was subjected to remove the adsorbed gases on the surface and the humidity by out gassed under vacuum at 300 °C for approximately 15 hours. After that, N_2 was purged to adsorb on its surface and analyzed adsorption isotherms at -196 °C.

      c. Leco TrueSpec CHN/O/S analyzer was used to measure the amount of carbon, hydrogen, nitrogen, sulfur and oxygen elemental composition of adsorbents. The oxygen content was estimated by difference assuming that only five elements, C, H, N, S and O, could be gasified from carbon samples.

      d. Thermo Scientific/Nicolet iS5 FTIR instrument was used to measure organic spectra. The spectrometer was collect 16 spectra in the range of 400-4000 cm^{-1} with a resolution of 4 cm^{-1}.

f. X-ray Photoelectron Spectroscope (XPS) was used to characterize surface chemistry of adsorbents. The samples were out gas under an ultra-high vacuum condition. Wide scan was performed at resolution pass energy 160 eV and narrow scan was performed at resolution pass energy of 20-40 eV and charge corrected to the main C1s peak at 284.6 eV.

g. Scanning Electron Microscope (SEM) was employed to study the surface morphology of activated carbon and treated activated carbon by using Hitachi TM3000, with an accelerating voltage of 15 kV and platinum coated under vacuum condition.

3.2.3 Adsorption and Desorption Measurement

The CO_2 adsorption and desorption performance of samples was evaluated in a thermogravimetric analyzer Netzsch F3 Jupiter. The procedure for evaluation the performance was shown below.

a. Around 10 mg of adsorbent was put in a crucible and placed into the STA analyzer.

b. The adsorbent was heated to 100 °C under 100 mL/min of N_2 flow and hold for 30 min to remove moisture.

c. The temperature was switched to the desire temperature at 5 °C/min and hold for 30 min with the same inert flow rate. Then a flow of 100 mL/min of CO_2 was introduced instead of N_2 to test the adsorption performance.

d. CO_2 was switched to N_2 at the same flow rate and temperature to test the desorption performance.

f. Finally, The temperature was cooled down under 100 mL/min flow of N_2.