

CHAPTER III

METHODOLOGY

3.1 Materials and Equipments

3.1.1 Equipment:

1. Thermogravimetric analyzer (TGA)
2. X-Ray Diffractometer (XRD)
3. Scanning Electron Microscope (SEM)
4. Surface Area Analyzer (SAA)
5. Mass Flow Controller
6. Humidity Analyzer
7. Mobile Electric Heater
8. Temperature Controller
9. Stainless tube and quartz Adsorber
10. Rotameter

3.1.2 Software:

1. Sigma Plot Program
2. Polymath . .
3. FORTRAN

3.1.3 Chemicals:

Adsorbents

- Activated Alumina
- Molecular sieve 1/8” and 1/16”

Gas

- Natural gas from PTTPLC
- Nitrogen (99.999%)

3.2 Experimental Procedures

3.2.1 Adsorbents Preparation:

Commercial adsorbents, activated alumina and molecular sieve zeolite type 4A of size 1/8" and 1/16", received from PTT Public Company Limited (PTTPLC) were prepared into deactivated forms with various extents of deactivation by hydrothermal steaming process. The activated alumina was kept in the hydrothermal steaming apparatus (Appendix I), which applied saturated steam at high temperatures of 400 to 550°C at a constant pressure of 1atm. Nitrogen was used as a steam carrier with constant flow rate 30ml/min. Two parameters, temperature and aging time, were varied along the experiment in order to obtain deactivated adsorbents with various extents of deactivation.

In case of the molecular sieve, another hydrothermal steaming technique was used to make the adsorbent deactivated. The water volume of 20 ml was fed into a stainless reactor at 260°C to generate supersaturated steam and adsorbed on molecular sieve of 20g. It was kept until the vapor evaporated out of the reactor. Aging time of this processing was used as the parameters to control various extents of deactivation.

3.2.2 Adsorbents characterization:

a. Static Adsorption Capacity (Chaikasetpaiboon, 2002)

The static adsorption capacity of adsorbents was evaluated by using a thermo gravimetric analyzer (TGA), the Dupont TGA 2590 equipped with a thermal analyzer 2000. The nitrogen gas was used as a reactant gas. The adsorbents were dried overnight in an oven at 110°C. Next, the adsorbents were kept in the humidity control box for saturation with water vapor for about 1 week. Then, the saturated adsorbents were tested for static adsorption capacity using TGA with the temperature program from 30 to 350°C and held at 350°C for 5 minutes. Before testing, the starting weight of each adsorbent sample was measured and recorded. Along the test, a derivative weight loss due to the increase of temperature was plotted, and the total weight loss in each temperature range was calculated, and then the static adsorption capacity was obtained.

b. Surface Morphology

The morphology of the adsorbents was examined by using the Scanning Electron Microscopy (SEM). The adsorbent was ground into very fine size and dried overnight in an oven at 110°C, then coated with gold, and placed in the SEM machine to determine the crystal size and the change of the morphology upon deactivation.

c. Surface Area Measurement (Lertviriyakijskul, 2000)

The surface areas of the prepared adsorbents were determined by the Brunauer-Emmer-Teller (BET) method. The characterization was based on the physical adsorption of nitrogen gas through the Sorptomatic 1990 at the liquid nitrogen temperature (77K). The quantity of gas adsorbed onto or desorbed from a solid surface at some equilibrium vapor pressure was measured. The change of surface area due to hydrothermal deactivation were analyzed and compared with undeactivated adsorbent.

d. Adsorbent Crystallinity (Chaikasetpaiboon, 2002)

The internal structure, bulk phase, and crystallinity of adsorbents were examined by the XRD technique. The XRD patterns were obtained by using a Rikagu X-Ray diffractometer system equipped with a graphite monochromator and a Cu tube for generating a CuK_α radiation. A sample was prepared by grinding adsorbents to very fine homogeneous powders and placed them over a glass slide holder. Then, the prepared sampled was scanned from 3 degree to 90 degrees (2θ) with scanning speed 0.02 degrees/min. The digital output of the proportional X-Ray detector, and the goniometer angle measurements were recorded. So, the change of the internal structure and composition of the crystalline material on the deactivated adsorbents was studied and analyzed.

3.2.3 Adsorption Isotherm

The adsorption isotherm of each prepared adsorbents was determined in an equilibrium mode. The experimental apparatus was designed and setup by Chaikasetpaiboon (2002) and Uttamaroop (2003). The apparatus mainly consists of a packed bed adsorber, and an auxiliary equipment for preparation of water-natural gas

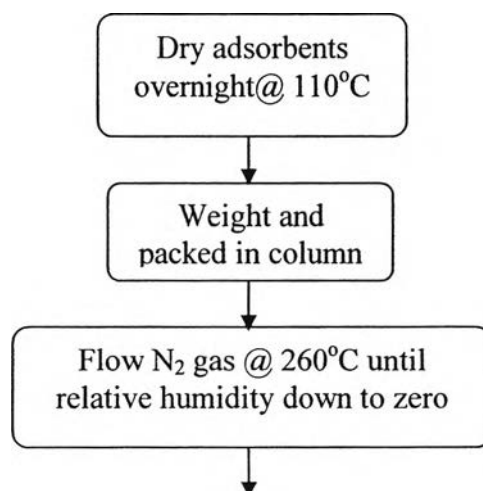
mixture, a gas mass flow controller, humidity analyzers and a mobile electric heater with a temperature controller. The experimental conditions are shown in Appendix C.

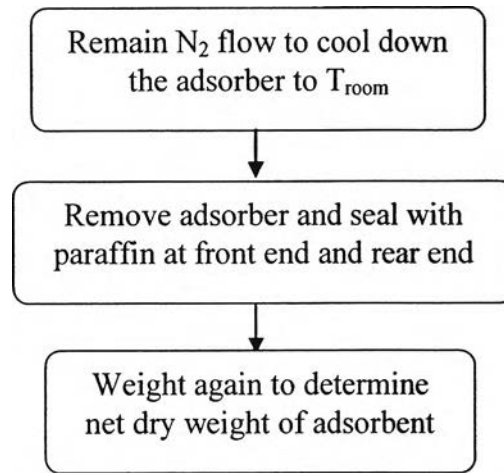
The stainless tube was used as an adsorber with the inside diameter of 1.7 cm and 10.0 cm height and the volume of about 22.7 ml.

The humidity of the feed inlet can be varied by adjusting the gas passing through a two-stage chamber, which contained the distilled deionized water inside, and another adjusted line is by-passed stream to get the expected humidity level. Also, the humidity of the saturated gas, the dry gas mixture at the inlet, and the gas stream at the outlet were measured by using a portable humidity analyzer, Cole Parmer Tri-Sense. Later on, since the portable humidity analyzers were not sensitive at low humidity, Uttamaroop (2003) improved the measurements by installing the CERMIT II hygrometer, Michell Instruments Ltd. in order to measure the humidity of the gas stream at low relative humidity. In addition, the nitrogen gas was used to attain humidity-free in the system, and the nitrogen flow rate was roughly controlled by using a rotameter.

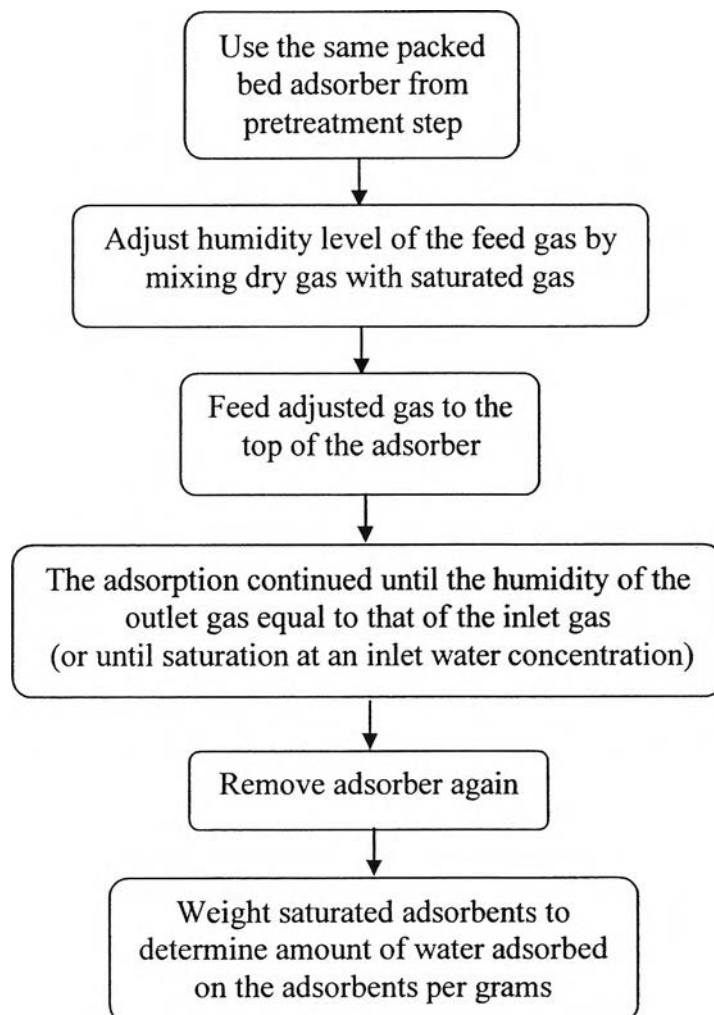
In this work the adsorption isotherm of each adsorbent was determined as the following experimental steps (Uttamaroop, 2003);

- Pretreatment step procedures followed this diagram below;





- Adsorption step procedures followed this diagram below;



The amount of water adsorbed on the adsorbents per gram was calculated as follows;

$$\text{Amount of water adsorbed} = \frac{(\text{Weight of saturated sample} - \text{Weight of dry sample})}{\text{Weight of dry sample}}$$

per gram of sample

In order to develop the adsorption isotherm curve, the ratios of the amount of adsorbed water on dry adsorbents were plotted versus the water inlet concentration or humidity.