CHAPTER III
METHODOLOGY

3.1 Materials and Equipment

3.1.1 Equipment:

The following equipment used in this work was obtained from the experimental set up constructed by Chaikasetpaiboon *et al.* (2002).

a. Gas mass flow controller

b. Humidifiers

c. Humidity analyzers

d. Adsorber (Pyrex)

e. Mobile electrical heater

f. Temperature controller

3.1.2 Materials:

a. Alumina

b. 4A molecular sieve with a pellet size of 1/8"

c. 4A molecular sieve with a pellet size of 1/16"

e. Natural gas

f. Nitrogen gas, 99.999% purity

3.1.3 Software:

FORTRAN language was used for numerically solving the theoretical breakthrough curve.

3.2 Experimental Procedures

3.2.1 Adsorption Isotherm

In this work, the adsorption isotherm of both fresh and deactivated adsorbents was investigated by following the work of Uttamaroop (2004).

3.2.1.1 Fresh adsorbents

a. Alumina, 4A molecular sieve 1/8", and a 4A molecular sieve 1/16" with an approximate weight of 5 grams were heated all night at 110°C to remove moisture.

b. The nitrogen gas was passed through the adsorbent at a temperature of 260°C until the relative humidity was down to zero to prepare the adsorbent.

c. The adsorber was cooled from 260°C to room temperature whereas nitrogen still flowed.

d. The weight of the adsorber after the drying step was determined before being put into the system again to perform adsorption mode.

f. In order to satisfy the humidity level, the feed gas was the mixture between dry gas and saturated gas to the desired value before feeding in the top of adsorber.

g. The feed gas was continually passed into the adsorber until the humidity level of outlet gas was equal to that of the inlet gas.

h. The weight of the adsorber was measured after when the adsorbent was saturated with water.

The amount of adsorbed water per weight of dry adsorbent can be calculated as follows:

(Weight of saturated sample – Weight of dry sample) / (Weight of dry sample)

The plot of this amount against the water concentration or humidity is the adsorption isotherm.

3.2.1.2 Deactivated adsorbents

In order to study the effect of deactivation on the adsorption isotherms, the following step was performed.

a. The 1/8" and 1/16" 4A molecular sieve were acceleratingly aged by boiling the adsorbent at 600°C for 45 minutes.

b. The adsorbents from Step1 with an approximate weight of 5 grams were heated all night at 110°C to remove moisture.

c. The nitrogen gas was passed through the adsorbent at a temperature of 260°C until the relative humidity was down to zero to prepare the adsorbent.

d. The weight of the adsorber after the drying step was determined before performing adsorption mode.

e. The wet gas was continually passed into adsorber until the humidity level of outlet gas was equal to that of the inlet gas

f. The adsorber was removed to find the new adsorption isotherm, as mentioned in the fresh adsorbents case.

The aging of the adsorbents was studied in different degrees of deactivation by varying the number of batches, and Steps 2 to 6 were repeated for each of the various degrees of deactivation.

3.2.2 Breakthrough Curve

The adsorption breakthrough curve of the process, including fresh and deactivated adsorbent can be obtained by applying the experiment steps as in Chaikasetpaiboon (2002):

a. The adsorbents, activated alumina, 4A molecular sieve 1/8" and 4A molecular sieve 1/16, were heated at 110°C to remove moisture.

b. The adsorbents from Step 1 were packed into the adsorber in the same ratio of actual operation at PTT public company.

c. Nitrogen gas was passed through the adsorbent at a temperature of 260°C until the relative humidity was down to zero, to prepare the adsorbent.

d. The adsorption step was performed by passing the desired humidified natural gas, after adjustment, to the top of adsorber. The outlet gas was measured by a humidity analyzer.

e. The outlet gas was continually measured until the humidity level at outlet was equal to the humidity level at inlet, and then the adsorption step was stopped. The breakthrough curve can be obtained by plotting the ratio of outlet water

to inlet water concentration ratio (C/C_0) versus time. The ranges of experimental conditions are shown in Table 3.1 below.

Table 3.1 Experimental conditions

Parameters	Value
Operating pressure (const.)	1 atm
Operating temperature (isothermal)	25°C
Humidity of natural gas feed	30% RH
Natural gas feed flow rate	458 ml/min
Contact time (based on feed flow rate)	9.83 sec
Bed volume	75 ml

3.3 Adsorbents characterization:

3.3.1 Static Adsorption Capacity (Khaikham, 2007)

In order to evaluate the static adsorption, the adsorbents were heated at a temperature of 110°C all night. And then they were saturated with water by putting them in the humidity control box. After that, each adsorbent was weighed before being tested using a thermogravimetric analyzer (TGA). The weight of each adsorbent was recorded a function of temperaturse from 30 to 600°C. The static adsorption capacity was the total weight loss in each temperature range.

3.3.2 Surface Morphology (Khaikham, 2007)

The adsorbents were made into very small sizes and dried overnight at 110°C. And then, those adsorbents were coated with gold before placing in the scanning electron microscopy (SEM) to investigate the crystal size and the changes of the morphology with varying deactivation.