CHAPTER III
EXPERIMENTAL SECTION

The experimental setup used in this work was constructed by Chaikasetpaiboon *et al.* (2002) to study the dynamic adsorption of water vapor from natural gas on multi-layer adsorbents. Three commercial adsorbents were packed in layers in the adsorber. The amount of adsorbents packed in the adsorber were the same volume ratio as the amount of adsorbents packed in the adsorber in dehydration unit of Gas Separation Plant (Rayong) of PTT Public Company Limited (PTTPLC). The operating temperatures for the adsorption and the regeneration were set at 25°C and 260°C, respectively. Adsorption and regeneration processes were studied under the same operating pressure of 1 atm.

3.1 Materials

Three commercial adsorbents, silica gel, 4A molecular sieve with the pellet size of 1/8" and 4A molecular sieve with the pellet size of 1/16", were packed in layers with the same volume ratio as in the commercial adsorption unit. They were obtained from PTT Public Company Limited (PTTPLC), and so was the natural gas sample. The highly purified nitrogen gas (99.999%), obtained from Thai Industrial Gas Public Company Limited, was used as a purge gas in the adsorbent pretreatment and regeneration steps. Moreover, the water vapor source, used for mixing with the natural gas in order to obtain a desired humidity level, was the deionized water.

3.2 Experimental setup

The experimental apparatus constructed by Chaikasetpaiboon *et al.* (2002) mainly consists of a gas mass flow controller, two humidifiers for mixing between water vapor and natural gas, humidity analyzers, an adsorber, and a mobile electrical heater with a temperature controller. The experimental flow diagram is shown in Figure 3.1.
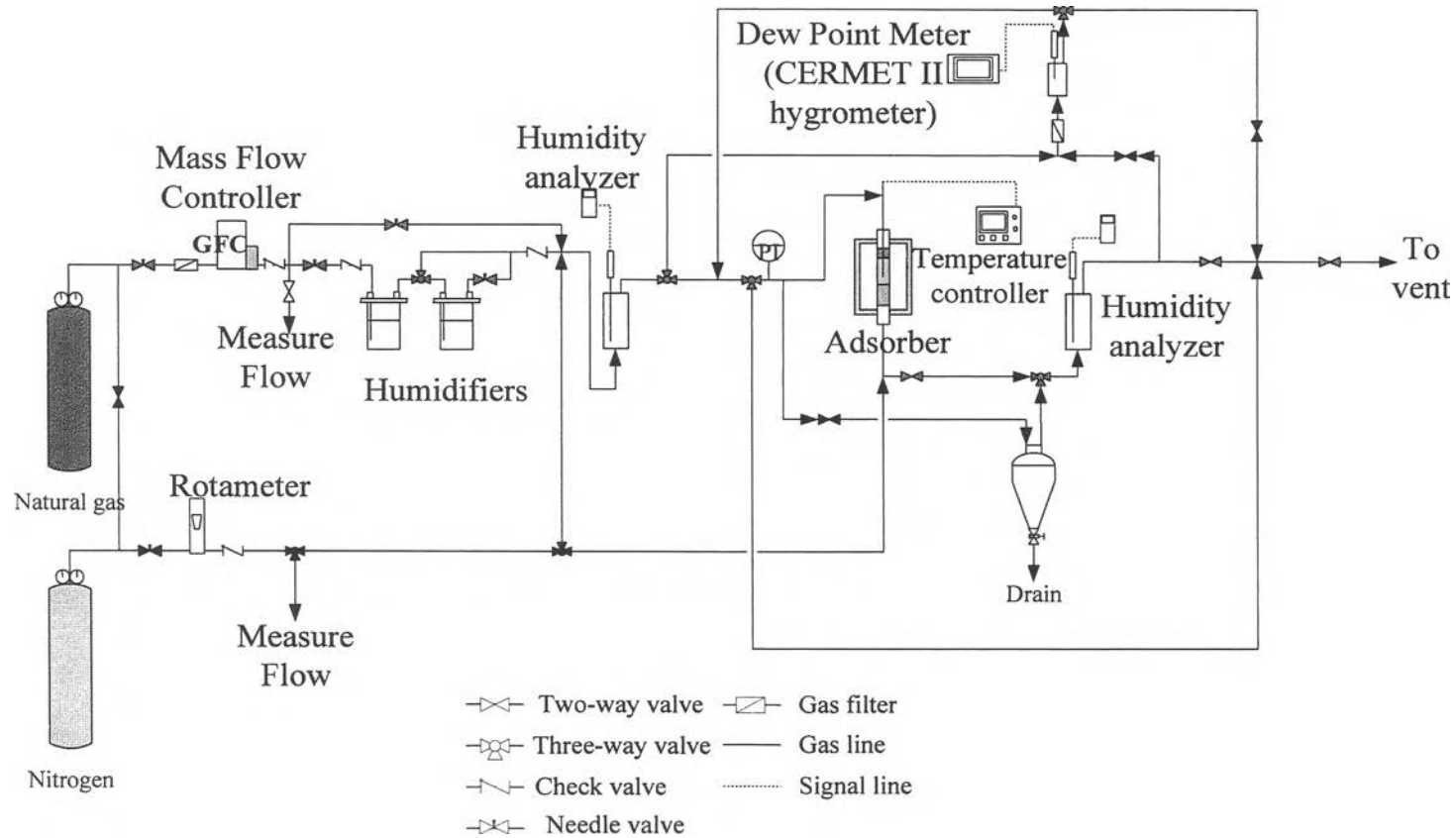
Figure 3.1 The schematic flow diagram for experimental setup (Chaikasetpaiboon *et al.*, 2002).

A gas mass flow controller (GFC), AALBORG Instrument & Controls Inc., model GFC171S with a range of 0 to 500 sccm as calibrated for methane gas was used to supply the constant volume flow rate of the natural gas. A micron filter was installed into the gas line before the mass flow controller. Check valves with 1 psig cracking pressure were also installed to the gas lines after the GFC to prevent reverse flow.

The humidity of the feed inlet can be varied by adjusting the flow of bypassed stream and the flow passed through a two-stage chamber which contained the deionized water. After that, the humidity of the saturated and the dry gas mixture was measured by using a portable humidity analyzer, Cole Parmer Tri-Sense® Relative Humidity/Air Velocity/Temperature Meter. The humidity of gas stream at the outlet from the adsorber was also measured by using the same model of the portable humidity analyzer as used at the feed inlet line. Since the portable humidity analyzers were not sensitive at low relative humidity, the CERMET II hygrometer, Michell Instruments Ltd., was also installed for measuring the humidity of the gas stream entering and leaving the adsorber.

The Pyrex tube with the inside diameter of 33 mm and a height of 120 mm, or volume of about 75 ml was used as the adsorber.

The mobile electrical heater was used in adsorbent pretreatment and regeneration steps. The temperature in the adsorber was monitored by using a J-type thermocouple, and was controlled to be constant by using the PID+FUZZY on the digital temperature controller, SIGMA model SF48 with a range of 25-300°C.

The nitrogen gas was used to attain humidity-free system and purge the system in the regeneration step. The nitrogen flow rate was roughly controlled by using a rotameter.

3.3 Experimental Procedures

3.3.1 Adsorption Isotherm

The adsorption isotherm of each adsorbent was determined as the following experiment steps:

(1) The adsorbent was dried overnight at 110°C, then weighted and packed in a column with the approximate weight of 5 grams.

(2) The pretreatment step was further performed with the flow of nitrogen gas at the temperature of 260°C until the relative humidity was down to zero.

(3) The nitrogen gas flow was remained to cool down the adsorber to the room temperature.

(4) The adsorber was carefully removed from the system and sealed with paraffin at the front end and at the rear end. After that, the net dried weight adsorbent was determined before resuming the adsorption step.

(5) The humidity level of the feed gas was adjusted by mixing dry gas with saturated gas to obtain the desired value before the feed gas was introduced to the top of the adsorber.

(6) The adsorption step continued until the humidity of the outlet gas was equal to that of the inlet gas.

(7) The adsorber was removed to measure the weight for saturated adsorbents.

The amount of water adsorbed on the adsorbents per grams could be calculated as follows:

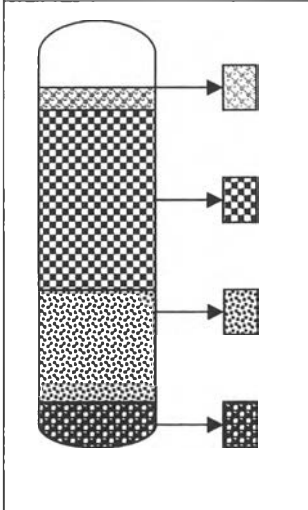(Weight of saturated sample – Weight of dry sample)/ (Weight of dry sample)

The adsorption isotherm was achieved by plotting the amount of adsorbed water per the weight of dry adsorbent versus the water concentration or humidity, which was obtained from running the adsorption at various humidity levels.

3.3.2 Experimental Breakthrough Curve

In order to obtain an adsorption breakthrough curve, the following experiment steps were executed (Chaikasetpaiboon *et al.*, 2002):

(1) All adsorbents, silica gel, 4A molecular sieve 1/8", and 4A molecular sieve 1/16", were dried at 110°C for overnight.

Table 3.1 Adsorber layout of the multi-layer adsorber employed in the adsorption study (Chaikasetpaiboon *et al.*, 2002)

Adsorber Layout	Adsorbent Type	Height (cm)	Weight (g)
	Silica gel	0.4	2.9643
	MolSiv (Zeolite) Type 4A with pellet size 1/8"	5.5	33.0121
	MolSiv (zeolite) Type 4A with pellet size 1/16"	2.9	20.6566
	Ceramic ball	*Inert material used as an adsorbent support	

Adsorber volume: 75 ml

(2) Each adsorbent was packed into the column (Pyrex tube) one by one as multiple layers according to designed volume ratios. The order of packing is shown in Table 3.1.

(3) The pretreatment step was performed with the same procedure as referred to the adsorption isotherm of water on multi-layer adsorbents section.

(4) The system was purged with N_2 gas until the humidity level was down to zero.

(5) A portion of natural gas was introduced through the humidifier chamber and mixed with the dry natural gas to obtain the desired humidity level, which was continuously detected using an on-line humidity analyzer.

(6) The humidified natural gas was passed to the top of the adsorber. The water content in the outlet gas from the bottom of the adsorber was immediately analyzed using an on-line humidity analyzer. The humidity values obtained from the humidity analyzer would be corrected by the humidity values obtained from the CERMET II hygrometer which was more sensitive at low relative humidity.

(7) The adsorption experiment was continuously performed, and the data were gathered until the adsorbents were totally saturated at the point where the water contents in the inlet and the outlet were equal.

(8) The adsorption experiment was stopped.

(9) The regeneration step was started with the same procedure as pretreatment procedure.

(10) The step (6) to (10) were repeated with different flow rates and/or humidity levels. The breakthrough curve could be obtained by plotting the outlet water and inlet water concentration ratio (C/C_o) versus time. The experimental conditions are shown in Table 3.2.

Table 3.2 The experimental conditions

Conditions	
Operating pressure	1 atm
Operating temperature (Adsorption step)	25°C
Humidity of natural gas feed - Adsorption isotherm - Breakthrough curve study	0-70 %RH 7%RH, 30%RH and 50%RH
Natural gas feed flow rate	130 and 260 ml/min
Contact time (Based on the feed flow rate)	17 and 34 sec
Bed volume (For breakthrough curve study)	75 ml