ETM0003

Experimental study of removal the humidity from humid gas by VSA process in a laboratory scale

Narit Lorpradit[1]and Yottana Khunathorn[1]

[1] Department of Mechanical Engineering, Faculty of Engineering, Chiang Mai University, Chiang Mai, Thailand, 50200
[*] Corresponding Author: E-mail: i_feelfree@hotmail.com, Telephone Number: +66-8-9644-5277

Abstract

Biogas can be upgraded into bio-methane and used in the vehicle as equivalent as NGV. Gas purification is a crucial step in biogas to bio-methane. There are many purification techniques, but pressure swing adsorption (PSA) is the most common and economical technique. However, the PSA cycle is used in research by many researchers for purification fluid. This research is focusing on the lab scale biogas to bio-methane purification, for used in vehicle replace NGV. This work studied about the purification technique by using vacuum swing adsorption (VSA) in the fixed-bed reactor. The working fluid used in this lab scale research is vapor water-nitrogen mixture, because the similarity of their atom size (N_2: 3.798 Å, CH_4: 3.758 Å). The fixed-bed reactor packed with carbon molecular sieve 4A was the adsorbent to adsorb the humidity from humidified nitrogen. The affects of operating parameters in experimental, which include cycle schedule and gas flow rate is 5 lit/min, pressure in adsorption is 3 bar, size of reactor are diameter 5 cm and long 100 cm, and the pressure drop is 0.075bar. The adsorption performance are 94.79% in first time but reduce after through desorption by the second is 89.84% and third is 87.30%.

Keywords: humidity removal, VSA, humid nitrogen.

1. Introduction

The present energy is important in transportation and logistics, which almost used oil. These are major energy and trend of the demand, have to increasing. For this reason the renewable energy are interest for replace oil. Thailand is agriculture country and has many wastes from farms. These wastes are used to produce the biogas with fermentation by anaerobic bacteria. Before using the biogas will be improve by remove other compounds until remain just methane then we call bio-methane [8]. The method is remove compounds from bio-methane will made bio-methane have a humidity that can't use in the engine. So we will be removed the humidity from bio-methane by technique VSA (Vacuum Swing Adsorption).

Many researches of PSA and VSA have received much attention. Developments of PSA and VSA gases separation are investigated, some parametric which effect to gas purity also have been studied [1, 2, 3, 5, 7]. The effect of pressure drop to fixed-bed [4], this depends on molar flow rate in the fixed-bed reactor.

Pressure/Vacuum Swing Adsorption are separation method by reactions between surfaces of adsorbents with molecules of adsorbate. Two reactions in these methods have 1.) Adsorption, this is a reaction for adsorb molecules of adsorbate by Van der Waals forces in pore of adsorbent at adsorbent's surface. 2.) Desorption, this reaction is in regeneration adsorbent process by used vacuum pump.

This research is find the behavior (life time, adsorb time and desorb or regeneration time) for removal humid from wet gas (Nitrogen) of fixed-bed reactor in laboratory. In this laboratory case study for explain to pilot scale and used to base for reduce the process in design the fix-bed reactor in produce dry gas in the future.

2. Theory and Methodology

This paper studies the physical adsorption of the fixed-bed reactor for removal the humidity from humid gas. This research is used nitrogen gas to replace bio-methane in laboratory scale because these have molecule size nearly. The reaction in fixed-bed reactor at removal humidity of wet gas, it is an adsorption between surfaces of adsorbent and molecule of water. This reacted by gas's diffusion inside porosity on adsorbent surface.

Adsorption is a reaction between surfaces of the adsorbents with molecule of the adsorbates. This reaction process is as follow: the molecules of adsorbate (gas or liquid) in mixing fluid, they will be adhere to the surface of the solid (adsorbent). At the surface area of the adsorbent has the pore and molecules of adsorbate will be adhere in this area by Van der Waals forces. Desorption is the reverse reaction of adsorption by this reaction is released the molecules of adsorbates from a surface of adsorbent. This reaction will be made a regenerate the adsorbent back to used again. These two reactions can expand in the diagram as follow:

$$\text{Adsorbent+Adsorbate} \underset{\text{Desorption}}{\overset{\text{Adsorption}}{\rightleftharpoons}} \text{Adsorption}$$

Or

$$\text{A+B} \rightleftharpoons \text{AB}$$

ETM0003

The adsorbent in this research is solid state or solid desiccant. The selection types of adsorbent will be considering from properties of each adsorbents. They are show in Table.1.

Table. 1 Show properties and type of carbon molecular sieve

Type	Typical Application
3A	Drying of unsaturated hydrocarbons. Cracked Gas Drying Drying of highly polar compounds, such as methanol and ethanol Drying of liquid alcohol with Z3-01 / 1-2 mm beads
4A	Drying and removing of CO2 from natural gas, LPG, air, inert and athmospheric gases, etc. Removal of hydrocarbons, ammonia and methanol from gas streams
5A	The strong ionic forces of the divalent calcium ion makes it an excellent adsorbent to remove water, CO2, H2S from sour natural gas streams, while minimizing COS formation. Light mercaptans are also adsorbed.
13X	Removal of CO2 and moisture from air and other gases. Separation of enriched oxygen from air. Removal of mercaptans and hydrpogen sulphide from hydrocarbon liquid streams (LPG, butane, propane etc.)

In this research is selected carbon molecular sieve 4A because its property is drying the natural gas from table.1. Then, they packed in to the fixed-bed reactor. The fixed-bed reactor will be design by calculation step from Gas Processors Suppliers Association (GPSA) [10]. By steps in the calculation size of the fixed-bed reactor has follows:

The first step is to determine the bed diameter by depends on the superficial velocity or V (The superficial velocity is the multiphase flow through the porous media). The pressure drop determined by modified Ergun equation. This relates the pressure drop to superficial velocity as follows:

$$\frac{\Delta P}{L} = B\mu V + C\rho V^2 \qquad (1)$$

Constants for Eq. (1) are:

Particle type	B	C
1/8"bead (4x8 mesh)	0.0560	0.0000889
1/8"extrudate	0.0722	0.000124
1/16"bead (4x12 mesh)	0.152	0.000136
1/16"extrudate	0.238	0.000210

From V value, it can be find to diameter in Eq. (2) by used $V_{\max}$ for selected the diameter of fixed-bed reactor in Eq. (3)

$$D_{\min} = \left(4q / \pi V_{\max}\right)^{0.5} \qquad (2)$$

$$V_{\max} = \left\{ \left(\Delta P / L\right)_{\max} / (C\rho) + \left[(B/C)(\mu/\rho)/2 \right]^2 \right\}^{1/2} - \left[(B/C)(\mu/\rho)/2 \right] \qquad (3)$$

The value of $(\Delta P / L)_{\max}$ in this equation depends on the sieve type, size and shape, but a typical value for design is 0.33 psi/ft [10]. In this research we selected the fixed-bed reactor has diameter 5 cm. Then, it will be calculated the new velocity for corresponding superficial velocity, $V_{adjusted}$ in Eq. (4) for diameter of reactor.

$$V_{adjusted} = V_{\max}\left(\frac{D_{\min imum}}{D_{selected}}\right)^2 \qquad (4)$$

From all equations, we have a limit range of pressure, flow rate and diameter and length of reactor. These have not effect to the reactor. These details as follow: maximum range for gas flow rate is 10 liter/min, minimum diameter is 3cm and pressure drop is 1.1psig (0.075 bar).

The next step is select period time in adsorption. The normally period times are eight to twelve hour. This research select time in adsorption is eight hour. The period time is a relationship with amount of remove humid from gas and amount of adsorbent to use per cycle. Then setup the experimental and recorded value of dry gas at outlet. The experimental has a condition as follow:

Fixed-bed reactor has a diameter 5 cm and long 100 cm. The wet gas (Nitrogen) flow through water with flow rate is 5 liters/min and pressure feed gas is 3 bars. In the period time is 8 hr.

3. Experiment setup

The experimental set up in laboratory scale is in Fig. 2. Then the adsorbents are saturated (can't adsorb water), they will be regeneration by vacuum pump for reused them again. Fig. 1 has schematic diagram of experiment setup in Fig. 2.

ETM0003

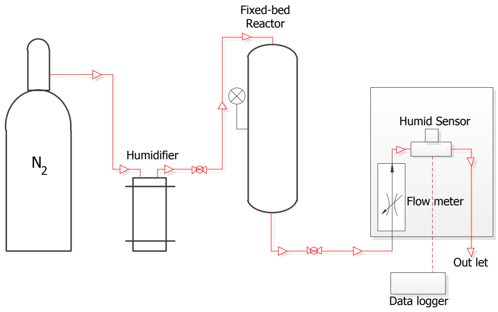

Fig. 1 Show the experiment setup schematic.

Fig. 2 Show the experimental set up.

This adsorption process is started by open the nitrogen gas tank flow through the humidifier first. The humidifier has water inside for give the humid to nitrogen gas. In the same time, we control the pressure and flow rate are 3 bars and 5 liters/min by ball valve and observe the pressure gauge. Then the humid nitrogen gas flow through the humidifier to fixed-bed reactor and recorded the value real time of drying nitrogen gas used the data logger. While the adsorbent cannot adsorb the humid we are stop this process and started to regenerate the adsorbent in desorption process. The desorption process is used the vacuum pump by close the valve (at before gas inlet the reactor) to made the reactor is close system and change at the bottom of reactor to connect the vacuum pump. The vacuum pump has specific: ¼ HP, flow rate 2 CFM and 1 state pump. It can made a pressure is -72 to -73 mmHg or around -0.1 bar.

Humid sensor used measure gas outlet used the dew point transmitter of TEKHNE TK-80. This sensor can measure gas humid in maximum pressure at 1MPa or 10bar and range of dew point are -80°C to 20°C.

4. The experiment result

The humid gas pass the humidifier measure has value average are 32.36% RH, moisture content 7.48 g/m^3. The adsorption experiment with this condition has a result shown in Fig. 3 for first adsorption.

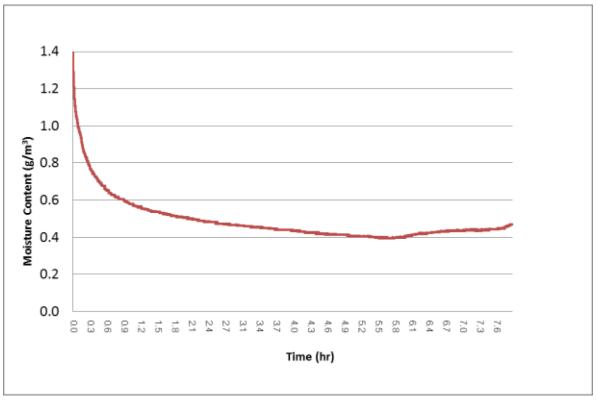

Fig. 3 Moisture content of drying gas in first adsorption

Fig 3 show the adsorption period in the reactor will adsorb humid a relatively stable to the adsorbents are saturated. These show in Fig 3 when the values of the moisture content are back to increased. Then we will stop the adsorption and made the regeneration (desorption) by used vacuum.

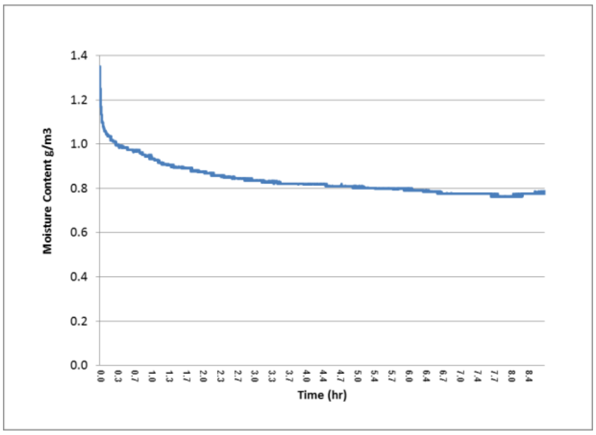

Fig. 4 Moisture content of drying gas in second times adsorption after vacuum first time

After vacuum adsorbents first time, they are adsorbed the humid again and they have decrease capacities in adsorbed humid. That is show in Fig 4, the value of moisture content in this adsorption have a less than the first adsorption in Fig 3. The values of moisture content are decreasing from 0.39 g/m^3 to 0.76 g/m^3 and the adsorbents are saturated.

Then the adsorbents are saturated in second adsorption. Desorption process will used again for regeneration the saturate adsorbents. In third adsorption has a result in Fig. 5. It showed the result of moisture content is 0.95 g/m^3. These results are showed the capacities in adsorb the moisture content in the humid gas by adsorbents in fixed-bed reactor.

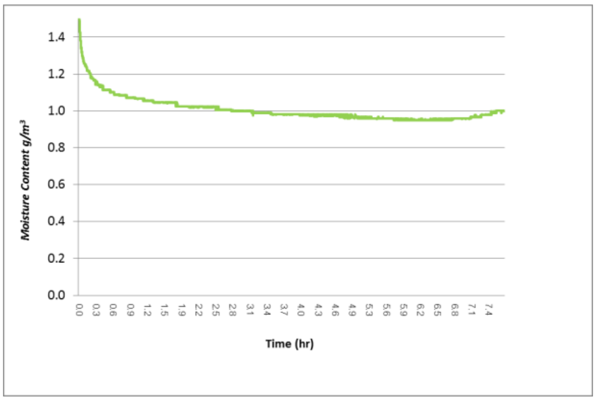

Fig. 5 Moisture content of drying gas in third times adsorption after vacuum second times

The comparison experimental results from adsorption three times are shown in Fig 6. The first adsorption can adsorb the most and can be reduce moisture content in gas to 0.39 g/m^3 at time around 6hr (5.77hr). After that, these adsorbents can't adsorb anymore or call saturation. Then, we made desorption process by vacuum and adsorb again. These can be two regeneration times because the adsorbents ability in adsorb humid is reduce obviously when they through the desorption process. Although time to saturation is longer than first adsorption but ability in adsorb humid less than by second adsorption has a moisture content is 0.76 g/m^3 even take over time more the first adsorption. Third adsorption has the least to adsorb amount humid and time to saturation.

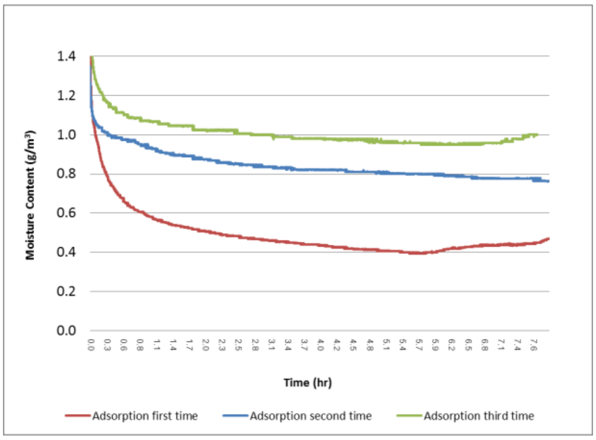

Fig. 6 Compare results of adsorption 3 times

Desorption process use period time is 8 hours for one regenerate adsorbents. If less than 8 hours the humid in adsorbents cannot removal all must be these adsorbents saturate faster and cannot removal humid from humid gas less than it should be.

5. Conclusions

In this work is study a behavior of carbon molecular sieve 4A with fixed the conditions and size of the fixed-bed reactor. The result is show the amount of humid to removal of adsorbents and this adsorb after regenerate adsorbent must be decrease. This value of decrease are show as follow: first adsorption has a max value is dry gas has a moisture content 0.39 g/m^3 but after desorption that value will decreed to 0.76 g/m^3 and 0.95 g/m^3. Although increase time to vacuum already, it has not effect to increase adsorb humid. The cycle of adsorbent from first adsorb the efficiency is 94.79% but decrease in second and third from desorption are 89.84% and 87.30%. This data from experiment will be found to isotherm and used to analyze inside the reactor with simulation program next.

6. List of symbols

B	constant in Equation (1) and (3)
C	constant in Equation (1) and (3)
D	diameter, cm
L	length of packed bed, cm
ΔP	pressure drop, psi
q	gas flow rate, m^3/min
V	gas velocity, m/s
μ	gas viscosity, centipoise
ρ	gas density, lb/ft^3

7. Acknowledgment

This work was sponsored by Energy Research and Development Institute Nakornping (ERDI-CMU), the Graduate School of Chiang Mai University, Energy Policy and Planning Office (EPPO) Ministry of Energy Thailand and the Mechanical Engineering Department and Thermo-Fluid Laboratory.

8. References

[1]. A.I.Fatehi, K.F.Lounghlin and M.M.Hassan (1995). Separation of methane-nitrogen mixtures by pressure swing adsorption using a carbon molecular sieve*, *Gas.Sep. Purif*. Vol.9, No.3, pp. 199-204, 1995

[2]. Krysztof Warmuziński and Wojciech Sodzawiczny (1999). Effect of adsorption pressure on methane purity during PSA separation of CH_4/N_2 mixtures, *Chemical Engineering and Process* 38, page 55-60.

[3]. R.Rajasree* and A.S. Moharir (2000). Simulation based synthesis, design and optimization of pressure swing adsorption (PSA) process, *Computers and chemical Engineering* 24 (2000), 2493-2505

[4]. M.H.Chahbani and D.Tondeur (2001). Pressure drop in fixed-bed adsorbers, *Chemecal Engineering Jurnal* 81, page 23-34

[5]. Salil U.Rege, Ralph T.Yang, Kangyi Qian and Mark A. Buzanowski (2001). Air-prepurification by pressure swing adsorption using single/layered beds, *Chemical Engineering Science* 56, page 2745-2759

[6]. Simone Cavenati, Carlos A. Grande and Alírio E.Rodrigues (2006). Separation of $CH_4/CO_2/N_2$ mixtures by layered pressure sing adsorption for upgrade of nature gas, *Chemical Engineering Science* 61 (2006), 3893-3906

[7]. E.Ryckebosch, M.Drouillon and H.Vervaeren (2011). Techniques for transformation of biogas to biomethane, *BIOMASS AND BIOENERGY* 35 (2011), 1633-1645

[8]. Carlos A.Grande (2012). Review Article Advance in Pressure Swing Adsorption for Gas Separeation, *International Scholarly Research Network ISRN Chemical Engineering*, Volume 2012, Article ID 982934, 13 pages

[9]. Brian Joseph Maring and Paul A.Webley* (2013). A new simplified pressure/vaccum swing adsorption model for CO_2 capture applications, International Journal of Greenhouse Gas Control 15, page 16-31

[10]. Gas Processors Association (2004): Gas Processors Suppliers Association Twelfth Edition FPS VERSION volumes I&II Sections 1-26, USA

[11]. W.J.Thomas and Barry Crittenden (1998): Adsorption Technology and Design. ISBN: 07506195967, Publication: April 1998, Elsevier Science & Technology Books