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Natural gas dehydration by desiccant materials

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Abstract Water vapor in a natural gas stream can result in line plugging due to hydrate formation, reduction of line capacity due to collection of free water in the line, and increased risk of damage to the pipeline due to the corrosive effects of water. Therefore, water vapor must be removed from natural gas to prevent hydrate formation and corrosion from condensed water.

Gas dehydration is the process of removing water vapor from a gas stream to lower the temperature at which water will condense from the stream; this temperature is called the ''dew point'' of the gas. Molecular sieves are considered as one of the most important materials that are used as desiccant materials in industrial natural gas dehydration.

This work shows a study of natural gas dehydration using 3A molecular sieve as a type of solid desiccant materials, the scope of this work was to build up a pilot scale unit for a natural gas dehydration as simulation of actual existing plant for Egyptian Western Desert Gas Company (WDGC). The effect of different operating conditions (water vapor concentration and gas flow rate) on dehydration of natural gas was studied.

The experimental setup consists of cylinder filled with 3A molecular sieve to form a fixed bed, then pass through this bed natural gas with different water vapor concentration, The experimental

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setup is fitted with facilities to control bed pressure, flow rate, measure water vapor concentration and bed temperature, a gas heater was used to activate molecular sieve bed.

Increasing water vapor concentration in inlet feed gas leads to a marked decrease in dehydration efficiency. As expected, a higher inlet flow rate of natural gas decrease dehydration efficiency. Increasing feed pressure leads to higher dehydration efficiency.

1. Introduction

1.1. Natural gas background

Natural gas is an important source of energy and is a naturally occurring fuel found in oil fields. Major gas fields were discovered in the 80s and 90s. The last 25 years have seen a remarkable growth in the contribution of gas to the world's total primary energy demand [1,2].

1.2. Natural gas hydrate

1.2.1. Hydrate properties

Hydrate formation is one of the problems that challenge the natural gas industry's move to high-pressure natural gas production, processing, and transportation. Hydrates are solid, crystalline compounds formed from water and hydrocarbon molecules such as methane and ethane.

They are very much like common ice in both their appearance and their properties with at least one important exception. Hydrates form at temperatures higher than 32°F, which is the freezing point of water [2].

1.2.2. Structure of hydrate

In a hydrate, the water molecules form a three-dimensional cage-like structure, with other molecule trapped within the cage. Examples of gases that form hydrates include methane, ethane, propane, butane, carbon dioxide, and hydrogen sulfide. These are all common components of natural gas. Only small molecules form hydrates typically, gases larger than butane does not form hydrates. The water molecules were referred to as the "host" molecules and the other compounds, which stabilize the crystal, are called the "guest" molecules, Fig. 1 shows water methane hydrate structure [2].

1.2.3. Hydrate conditions

The formation of a hydrate requires the following three (necessary) conditions:

(i) Lowering gas temperature.
(ii) Elevation of gas pressure.
(iii) The natural gas composition {Water vapors "host" and hydrocarbon percentage "guest"}, a sufficient amount of water should not too much, not too little and free water is not required.

It is necessary to prevent the condensation of liquid water and hydrocarbons to ensure trouble free operation of a natural gas transmission system. Apart from the risk of hydrate formation, is the liquids can reduce the volumetric capacity of the system and interfere with the operation of pressure regulators and filters. Condensed liquids accumulated in pipelines, caused an increase in operating pressures and potential damage to equipment due to liquid carryover [3–5].

1.3. Technologies used for dehydration

There are different techniques employed for dehydrating natural gas, but only two types of dehydration techniques are commonly in current use: absorption by liquid desiccants and adsorption by solid desiccants [6].

Adsorption (or solid bed) dehydration is the process where a solid desiccant is used for the removal of water vapor from a gas stream. The solid desiccants commonly used for gas dehydration are those that can be regenerated and, consequently, used for several adsorption–desorption cycles [6].

1.4. Types of solid desiccants

Three basic materials are used most commonly because they possess these characteristics in a satisfactory manner:

(i) Activated alumina. (ii) Silica gel and silica-alumina gel. (iii) Molecular sieves.

1.5. The dynamics of adsorption bed

Fig. 2 illustrates the basic behavior of an adsorbent bed in gas dehydration service. During normal operation in the drying (adsorbing) cycle, three separate zones exist in the bed:
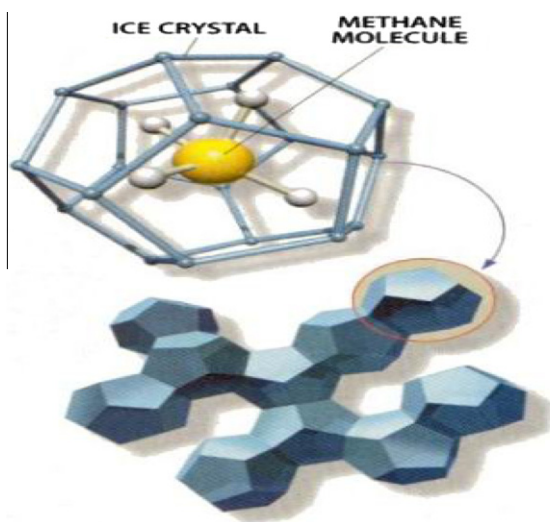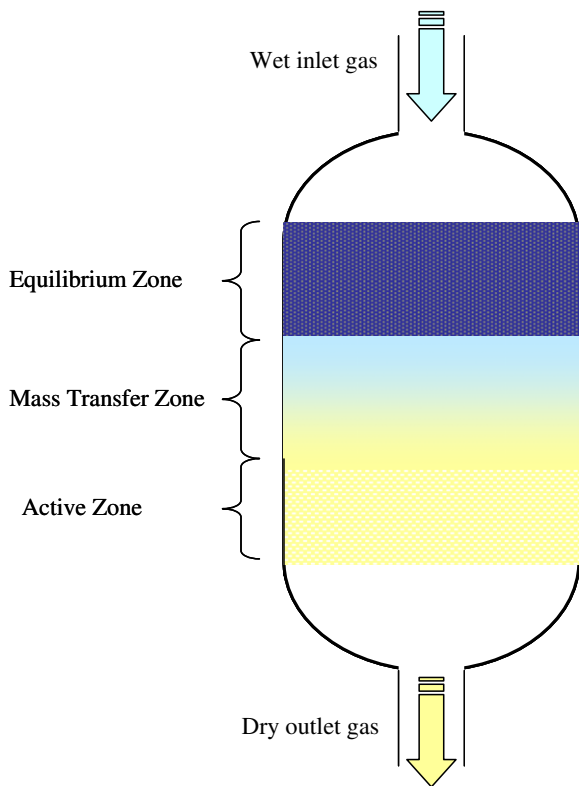

Figure 1   Water methane hydrate structure.

Figure 2 Three zones of adsorption.

(i) Equilibrium zoneIn the equilibrium zone, the desiccant is saturated with water; it has reached its equilibrium water capacity based on inlet gas conditions and has no further capacity to adsorb water.

(ii) Mass transfer zone (MTZ)Virtually all of the mass transfer takes place in the MTZ, a concentration gradient exists across the MTZ.

(iii) Active zone

In the active zone the desiccant has its full capacity for water vapor removal and contains only that amount of residual water left from the regeneration cycle. When the leading edge of the MTZ reaches the end of the bed, breakthrough occurs [6].

1.6. Aim of the work

The scope of this work was to build up a pilot scale unit for a natural gas dehydration using 3A molecular sieve as a type of solid desiccant materials to make a simulation of actual existing plant conditions of Western Desert Gas Company (WDGC).

The experimental setup consisted of cylinder filled with 3A molecular sieve to form a fixed bed, then natural gas with different water vapor concentrations was pass through this bed. In addition to that, there are facilities to control bed pressure, flow rate, measure water vapor concentration and bed temperature. Also gas heater was used to activate the molecular sieve bed. The effect of different operating condition such as water vapor concentration and gas flow rate on dehydration of natural gas, was studied in the present work.

2. Review of literature

The effectiveness of parameter of water adsorption on molecular sieve was investigated to find optimum operating conditions. The obtained experimental breakthrough curves were fitted to theoretical models in order to establish the main mechanisms of mass transfer. The significant dispersion values are also proven by the Peclet number obtained for each breakthrough curve. Molecules with large polarity can be adsorbed preferentially under identical conditions. Molecular sieves exhibit intraparticular diffusion, which is specifically controlled by molecular diffusion [7].

Water adsorption in zeolites is based on physisorption. The main driving force for adsorption is the high polar surface within the pores. This unique characteristic distinguishes zeolites from other commercially available adsorbents, enabling an extremely high adsorption capacity for water vapor and other polar components even at very low concentrations. Molecular sieves contain a uniform network of crystalline pores and empty adsorption cavities. Because of its uniform structure; molecular sieve will not give up moisture into the package as temperatures rise [8].

Experimental data for dynamic adsorption of ethyl acetate present in air using 5A and 13X molecular sieves are generated with variation of inlet adsorbate concentration, inlet velocity and bed diameter to bed length ratio to study their effect on the adsorber bed performance. On decreasing inlet velocity, breakthrough occurs late and almost a proportionate increase in breakthrough time was noted with decrease in inlet velocity (constant pattern behavior is achieved) [9].

The primary effect of inlet velocity is on the rate of movement of mass transfer zone (MTZ). The movement of MTZ is directly related to inlet flow velocity. The similar nature of the breakthrough curves in case of both the sieves indicates that mass transfer rate is not sensitive to inlet velocity. The constant pattern breakthrough curves for both the molecular sieves (5A and 13X) with change in inlet velocity indicate that mass transfer rate is controlled by pore diffusion and not by external film [10].

The insignificant temperature rise in case of 5A may be due to comparatively less saturation adsorption capacity (poor adsorption characteristic) of the EA-5A molecular sieve system. Lower saturation adsorption capacity reduces the rate of adsorption to give lower temperature rise, which becomes further low due the convective transport of heat with the fluid.

The equilibrium adsorption capacity of Ethyl acetate in 13X is higher and it behaves closer to ideal behavior than 5A and mass transfer zone is well contained within the bed due to higher affinity of the sieves for Ethyl acetate [11].

For a long bed, the mass transfer zone will be contained within the bed. In such cases the concentration breakthrough profile will remain unchanged with change in L (constant pattern behavior). Mathematical model based on Linear Driving Force approximation is validated with experimental data and simulated to understand effects of other pertinent parameters such as overall mass transfer coefficient, saturation capacity and bed to wall heat transfer coefficient. 13X molecular sieves with higher capacity than 5A shows closer to ideal adsorption behavior. Mass transfer rate is controlled by pore diffusion [12].

Inverse gas chromatography was used to evaluate the adsorption thermodynamic parameters (ΔH, ΔS and ΔG) of some probes, each representing a class of organic compounds (n-hexane, cyclohexane and benzene) on some adsorbents (activated alumina, and molecular sieves 3A and 5A). It was found that benzene exhibits more negative ΔH than for n-hexane and cyclohexane on molecular sieves 3A, 5A and alumina. In addition, interactions of the benzene with molecular sieves 3A and 5A was found stronger to be than that on alumina. On the contrary, interactions of the hexane with alumina were found to be stronger than those on molecular sieves 3A and 5A [12].

Dehydration of ethanol via adsorption using molecular sieves has been recently suggested as a promising alternative to the conventional separation methods for ethanol–water mixtures. 3A zeolites possess selective micro pores whereon, due to the small size of their pores, the water molecules are adsorbed while the ethanol molecules are excluded. The thermodynamic results showed that the spherical and cylindrical zeolites have the same adsorbing capacity at the temperatures studied and that this capacity decreases with temperature increase. The Langmuir isotherm satisfactorily correlated the experimental data for the experimental temperatures and concentrations studied. The kinetic tests are dependent on the parameters studied, where a temperature increase resulted in an increase in the diffusivity values for both zeolites. The diameter increase of the spherical particles resulted in a decrease in the adsorption rates, but the diffusivity remained constant with a mean value of $1.67.10^{-5}$ cm^2/s [13].

From the analysis of the regeneration of weak desiccants, it was found that the higher the regeneration temperature, the smaller are the required quantities of regeneration gas. For solid desiccants as silica gel; the effects of the pressure, temperature, and quantity of purge gas on the degree of drying are studied. In all of the experiments, the drying operation gave dew points of minus 30 °C or lower (pressure 20 MPa). With a regeneration temperature equal or close to the adsorption temperature, the quantity of purge gas depends on its pressure; when the regeneration pressure is increased from 0.1 to 0.5 MPa, the quantity of purge gas is increased from 10% to 20%. Regeneration of the sorbent at a temperature above the adsorption temperature leads to a reduction of the purge gas quantity. The higher the regeneration temperature, the less the required amount of purge gas. If the regeneration temperature is increased to 70–80 °C, the quantity of purge gas could be reduced by 3%; In this case, the regeneration pressure is not an important factor [14–16].

The design and analysis of a two-tower, silica gel dehydration unit to dry one million standard m^3 of natural gas per day and the effects of various operating parameters on the design of the unit was studied. Calculations are made for the design of this, solid desiccant dehydrator using silica gel as one of the most versatile solid desiccants for the dehydration of natural gas.

It is proved that the operating temperature of the gas has negligible effect on the dryer shell thickness but it has considerable impact on the desiccant mass required for dehydration. As the operating pressure of the gas increases, the desiccant mass required for dehydration decreases but the rate of decrease is insignificant for pressures greater than 6 MPa.

As the pressure increases, the desiccant bed diameter decreases whereas the bed length increases. It was noted that the changes in desiccant bed diameter is insignificant compared to the changes in the bed length, the gas velocity affects both diameter and length of the bed. As the superficial gas velocity increases, diameter decreases and length increases.

As the gas flow rate increases, the required desiccant volume for dehydration increases and the ratio of (bed length/bed diameter) decreases, and as the pressure increases the ratio of (bed length/bed diameter) increases [17,22].

3. Materials and methods

3.1. Materials used

3.1.1. Molecular sieve and ceramic balls
In this experiment molecular sieve type 3A was used (Size of MS pellets: 3.2 mm). Table 1 shows the specification of molecular sieve type 3A used in experiment. This type of molecular sieves was supplied by Specialty Chemicals Actor (CECA) Company [France]. In addition, ceramic balls 1/2 inch supplied by CECA Company [France] was used as thermal chock absorber for bed packing in this experiment.

3.1.2. Gases used
Two types of gases were used in experiment:

Natural gas.
Nitrogen gas.

Table 2 shows the composition of natural gas used, and Table 3 shows the specification of natural gas used.

3.2. Apparatus and method of measurements

3.2.1. Dew point temperature measurements
In this experiment drager tube was used (type: Water vapour 5/a-P, the supplier: Drager Rohrchen) to determine water dew point for natural gas in which it measure the water vapor concentration in ppm.

3.3. Experimental setup

3.3.1. Parts of adsorbing bed

(i) The adsober bed
   It is made from carbon steel in the form of a cylinder (Length × Diameter; 1070 × 125 mm).
(ii) Bed packing
   A mesh screen (slot diameter 2 mm) support was installed in lower part of the cylinder, two layers of 6.35 mm (1/2 inch. diameter) ceramic balls were introduced in the cylinder, then 6.1 kg of molecular sieve type 3A (size of MS pellets: 3.2 mm) (N.B: the diameter of molecular sieves bed is 125 mm and its height 640 mm), after that two layers of 6.35 mm (1/2 inch.) ceramic balls were introduced, finally a cover of mesh screen (slot diameter 2 mm) was installed in upper part of cylinder.
(iii) Pipes network and valves
   (a) The pipes network
       The pipe network consisted of 1/4 inch 316 stainless steel, and sealant (type LOXEAL 18-10 pipe seal-

Table 1 Properties of 3A molecular sieves used.

Subject	Descriptions
Type of crystal structure	Cubic
Molecular formula	$0.45K_2O \cdot 0.55 \ Na_2O \cdot AI_2O_3 \cdot 2 \ SiO_2 \cdot n \ H_2O$
Tapped bulk density, EN ISO 787-11	$750 \ kg/m^3$
Nominal pore diameter	3 Angstroms
Bead size (nominal)	2–3 mm
Equivalent (approx.)	8×12 mesh
Crushing strength	45 N
Equilibrium water adsorption capacity, 45 mbar at 25 °C	20% by Wt
Residual water content, 550 °C as shipped	1.5%
Specific heat (approx.)	1.07 KJ/Kg °C
Heat of adsorption	4200 KJ/Kg water
Regeneration temperature recommended	200–230 °C
Solubility in water	Insoluble
pH	9.0–11.5 for 50 g/L H_2O as suspension at 20 °C

Table 2 Composition of the natural gas used.

Components	Mol%
Nitrogen	0.659
Carbon dioxide	3.836
Methane	80.540
Ethane	9.666
Propane	3.703
I-Butane	0.548
N-Butane	0.762
I-Pentane	0.127
N-Pentane	0.094
Neo-Pentane	0.001
C_6^+	0.059

Table 3 Specifications of natural gas used.

Gas specifications	Value
Gross heating value (G.H.E) at 1 atm. 60°F (BTU/SCF)	1143.983
Molecular weight	20.38
Density at 1 atm. 60°F (Kg/m^3)	0.860
Relative density (R.D) at 1 atm.60°F	0.703

ant) was used to prevent gas leakage from fitting, this pipe was fitted to achieve the following targets:
- Purging of the whole system.
- Activation of molecular sieve bed.
- Dehydration of natural gas during runs of the experiment.

(b) Valves used

Two types of valves were used:
- 316 Stainless steel throttling valves to reduce the gas pressure, and control the pressure at sampling point and at the outlet points.
- 316 Stainless steel 1/4 inch ball valves to direct the gas flow and to control gas flow. Fig. 3, shows drawing of the experimental setup.

Fig. 3 shows drawing of the experimental setup, Fig. 4 shows flow direction and layers in activation and adsorption steps.

3.4. Natural gas adsorption runs

The following variables were studied during experimental work:

(i) The variation of gas flow rate (range: $50 \approx 200$ L/h).
(ii) The variation of inlet water vapor concentration (range: $340 \approx 550$ ppm).

The following properties were measured during experimental work:

(i) The outlet water vapor concentration.
(ii) The temperature of bed layers at four different locations.

4. Results and discussion

4.1. The effect of gas flow rate on the removal of water vapor during adsorption

Fig. 5 shows the variation of percentage removal of water vapor for different natural gas flow rates during water adsorption. It is clear from fig. 5, that at the start of water vapor adsorption the percentage removal of water vapor is low then increases gradually until it reached a constant value. This phenomena was repeated for various flow rates. This can be attributed to The fact that adsorption is governed by pore diffusion mechanism (Physisorption), since the run was carried out at low pressure ($2 \ Kg/cm^2$) and hence low driving force which increased the period to reach the maximum water adsorption capacity.

It is also noted that as the inlet gas flow rate increases (from 50 to 120 L/h) the removal percentage of water vapor decreases. This can be attributed to the increase in water load as the gas flow rate increases and decrease of contact time between gas and molecular sieves.
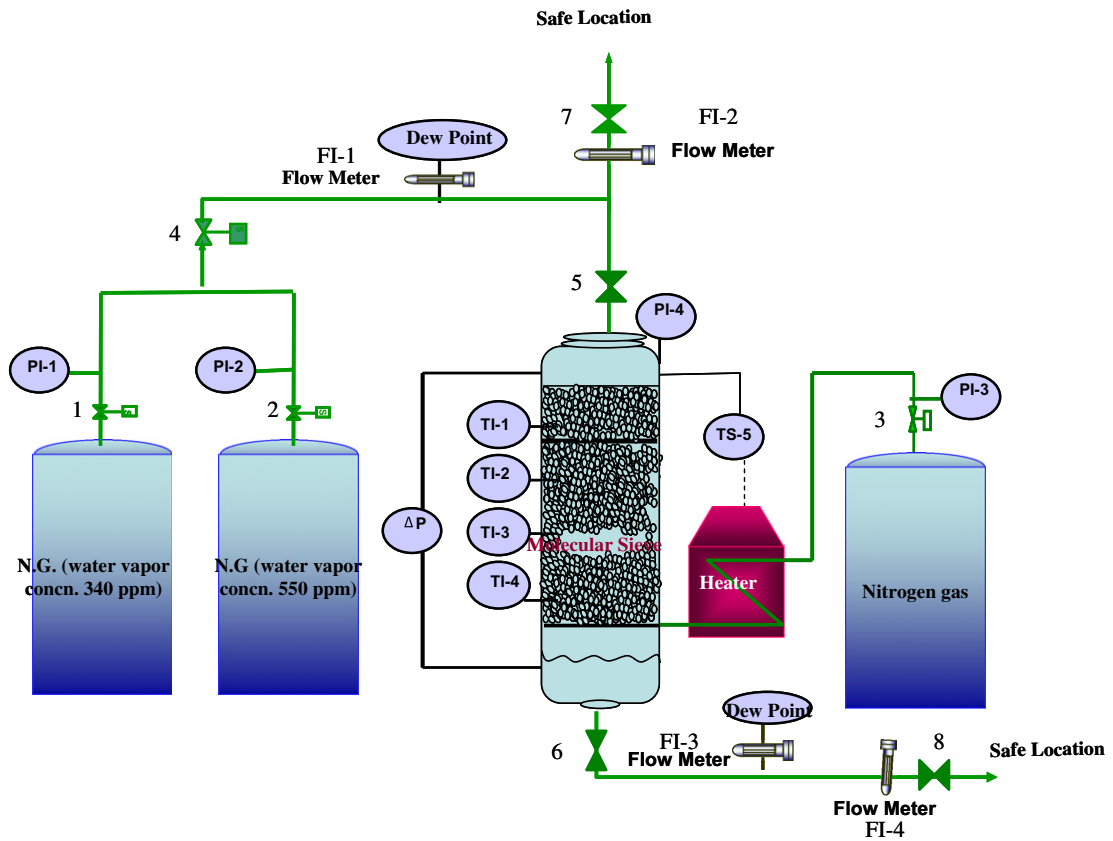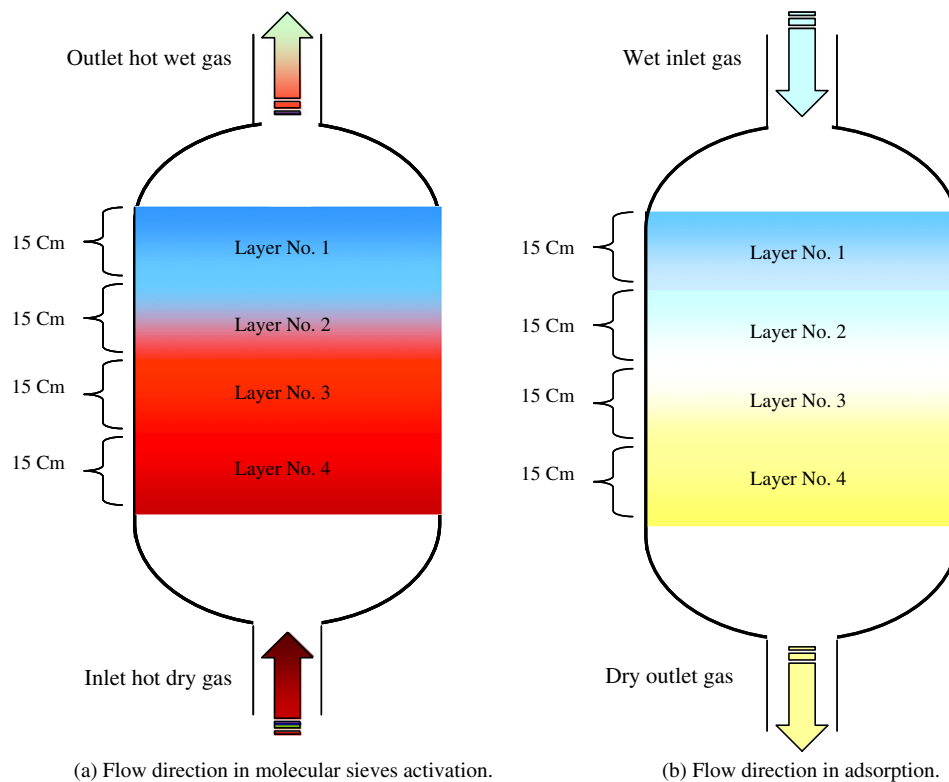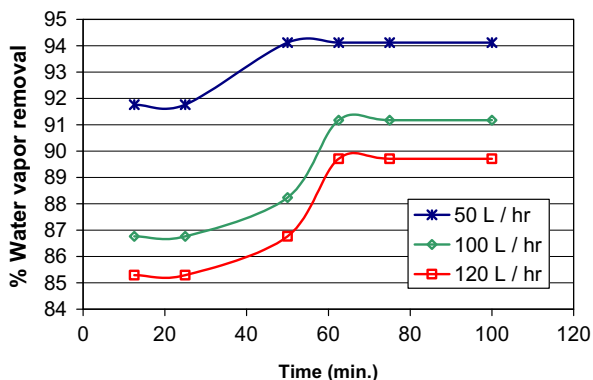
Figure 3   Experimental setup.

(a) Flow direction in molecular sieves activation.   (b) Flow direction in adsorption.

Figure 4   Flow direction and layers in activation and adsorption steps.

Figure 5 Effect of gas flow rate on the percentage removal of water vapor with time (inlet gas water concentration 340 ppm).

Fig. 6 shows the variation of maximum removal percentage of water vapor for different natural gas flow rates during bed water adsorption. It is clear that as the gas flow rate increases the maximum removal percentage of water vapor decreases. This can be attributed to the decrease of the residence time of the gas as its flow rate increases.
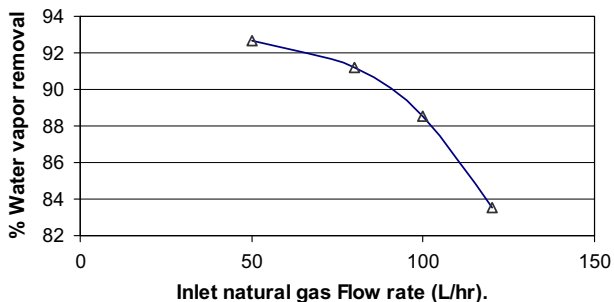
4.2. The effect of inlet water vapor concentration on removal percentage

Fig. 7 shows the variation of percentage removal of water vapor with various inlet water vapor concentrations, (340 and 550 ppm). It is clear from Fig. 6, that the percentage removal of water vapor increases with increases the inlet water vapor concentration. This can be attributed to the increase of driving force as the inlet water vapor concentration increases. It is also noted that the removal percentage of outlet water vapor increases with decreases inlet flow rate. This can be attributed to the decrease of the residence time of the gas as its flow rate increases.

4.3. The effect of inlet water vapor adsorption on the bed temperature distribution

4.3.1. Inlet flow rate 50 L/h & concentration 340 ppm

Fig. 8 shows the variation of bed layers temperature with time during adsorption. It is clear from Fig. 8, that at initial time of adsorption the temperatures of layers were low and approximately have the same value. Then temperature increases with

Figure 6 Effect of gas flow rate on the maximum percentage removal of water vapor (inlet water vapor concentration 340 ppm).
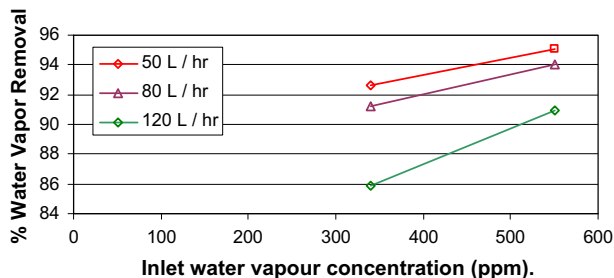
**Figure 7** The variation of water vapor percentage removal with inlet water vapor concentration.

time during adsorption process. This can be attributed to the fact that water adsorption is an exothermic [1–5] process, so temperatures increases as water quantity adsorbed increases (so heat liberated increases), After that the temperatures reached a maximum value which corresponds to the maximum heat liberated from adsorption.

Fig. 9 shows variation of temperature along bed at blank run and after 100 min from starting water vapor adsorption (N.B.: Blank run was just passing dry gas (nitrogen) without adsorption before starting adsorption run). It is clear from Fig. 5, that temperature was high at layer No. 1 and decreases gradually to lowest temperature at layer No. 4. Also it is noted that the temperature difference between blank run (without adsorption) and after 100 min was high at layer no.1 and low at layer No. 4. This can be attributed to the fact that layer No. 1 was in the front of adsorber bed so it has removed large quantity of water, hence its temperature increases with increase of heat liberated from water adsorption.

It is also noted from Fig. 9, that in blank run there was slight variation of layers temperatures.

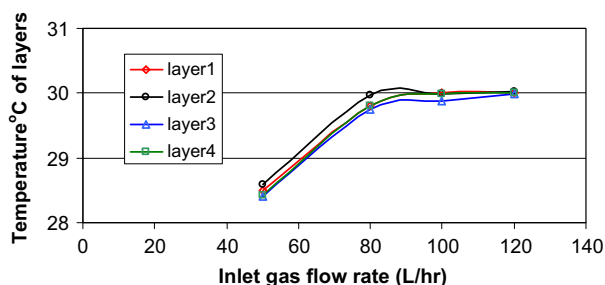### 4.4. The effect of inlet gas flow rate on temperature of the layers

Fig. 10 shows the variations of maximum bed layers temperatures with inlet gas flow rate (inlet water vapor concentration 340 ppm). It is clear from Fig. 10, that at low flow rate the temperature of layers is low then temperature increases with increasing gas flow rate as a result of increase of water quantity adsorbed so heat liberated increases, till reach to a maximum temperature.
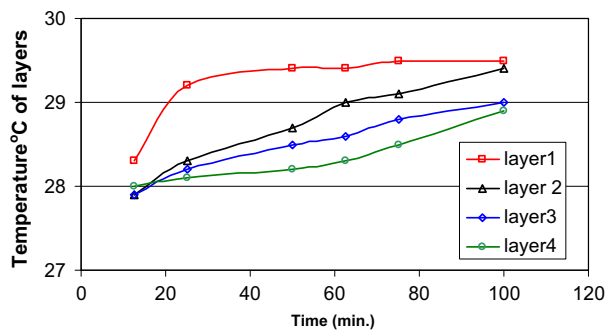
Figure 8 Variation of bed layers temperature with time during adsorption (inlet concentration 340 ppm & 50 L/h).
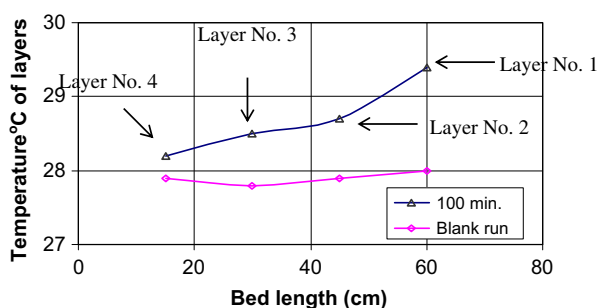
Figure 9 Variation of temperature along bed at blank run and after 100 min from adsorption (inlet concentration 340 ppm & 50 L/h).

Figure 10 The variations of maximum bed layers temperature with inlet gas flow rate (inlet concentration 340 ppm).

5. Conclusions

The present study on dehydration of natural gas using molecular sieves (3A) revealed the following conclusions:

1. Increasing the gas flow rate decreases the percentages of water vapor removal.
2. Increasing the inlet gas water content increases the percentages of water vapor removal.
3. Increasing the wet inlet gas flow rate increases the beds temperature up to certain value after which the beds temperature remained constant.
4. In case of high concentration of water in the natural gas (550 ppm) increasing the flow rate beyond 150 L/h led to further increase of beds temperature.
5. For water content of 340 ppm and up to a flow rate 80 L/h there was slight difference between beds temperature of blank runs and these during dehydration. Increasing gas flow beyond 80 L/h led to higher difference between temperature of beds in blank runs and these during dehydration.
6. For water content of 550 ppm and up to a flow rate 120 L/h there was a slight difference between beds temperature of the blank runs and these during dehydration. Increasing flow rate beyond 120 L/h led to higher difference between temperature of beds in blank runs and these during dehydration.
7. For a very high gas flow rate 200 L/h and high water content (550 ppm) the difference between temperature of beds at blank runs and the two layers at the end of the fixed

bed was higher than that the two layers in the front of the beds.

6. Recommendations

Further study to investigate the effect of type of molecular sieve, percent CO_2 and H_2S on dehydration efficiency.

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