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Reflux in a gas dehydration plant

Gas dehydration by adsorbent processes may lead to the damaging regeneration reflux phenomenon during adsorbent regeneration

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Dehydration of natural gas entails the removal of water that is associated with natural gases in vapour form. The natural gas industry has recognised that dehydration is necessary to ensure smooth operation of gas transmission lines. This pretreatment prevents the formation of gas hydrates and reduces corrosion. The three major methods of dehydration are direct cooling, adsorption and absorption. Adsorption-based processes for separation of multi-component gaseous mixtures are becoming increasingly popular. The new generation of synthetic and more selective adsorbents developed in recent years has enabled adsorption-based technology to compete successfully with traditional gas separation techniques.

Any adsorption-based separation process requires two essential steps: adsorption during which one or more components are preferentially adsorbed/separated; and regeneration during which these components are removed from the adsorbent bed. The adsorbent is repeatedly used in cycles by carrying out these two steps. When a regeneration step is carried out through reduction of the total pressure, the process is called pressure swing adsorption (PSA). Temperature swing adsorption (TSA) is another technique used for regenerating a bed of adsorbent that is loaded with the targeted impurity gas. This technology began commercially in the 1960s and continues today for drying continuous air and natural gas as well as other purification applications such as carbon dioxide stripping from air. TSA exploits the capacity of certain adsorbent materials, such as activated alumina, silica gel and zeolites, to adsorb gases at moderate temperatures (40°C, 100°F) and later release them when the temperature rises above 120°C (250°F).

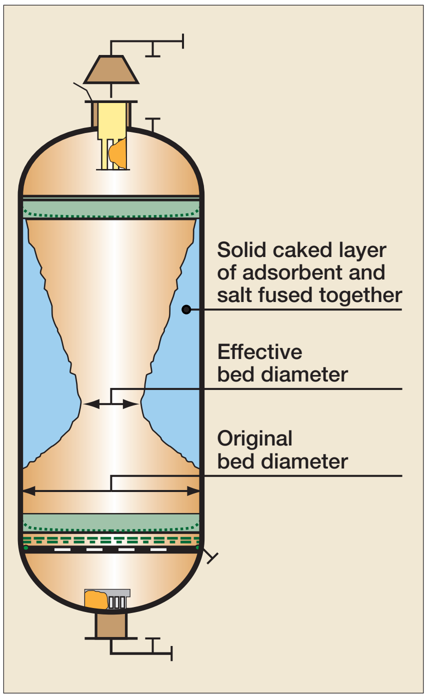


Figure 1 Schematic of a bed faced with regeneration reflux

Natural gas treating units using molecular sieves and TSA technology are usually optimised by manipulating both the adsorption and the regeneration time. By reducing the adsorption time, both the vessel size and the amount of adsorbent used are reduced. Therefore, the total cycle time is usually designed such that at the end of the adsorption a short time is available for appropriate regeneration of the adsorbent. Hence, the inlet section of the adsorption bed is faced immediately with a high temperature from the start of the regeneration without any heating ramp. Heating up the adsorber without using a heating ramp causes a strong temperature difference in the bed. So, at the bottom, the molecular sieve is very hot and desorbs the adsorbed water while the top layers are still at adsorption (low) temperature. Therefore, water desorbed in the bottom layer condenses in the top layer. This phenomenon is called refluxing or retro-condensation. A schematic diagram of an adsorber with regeneration refluxing is shown in **Figure 1**. To prevent this catastrophic phenomenon, a good molecular sieve formulation (binder and zeolite) or improvement in the regeneration condition is inevitably required.

In this article, modelling of the regeneration reflux phenomenon during regeneration is performed and the effects of it on the adsorption process are reviewed. Recommendations to prevent this phenomenon in a commercial scale dehydration unit (as a case study) are presented.

Process description

The purpose of a natural gas dehydration package is to reduce the water content of the natural gas to avoid freezing and hydrate formation in the pipeline. In order to utilise natural gas for urban consumption, the water dew point should be reduced to below -10°C, accomplished by using a molecular sieve adsorption unit which adsorbs water from the inlet gas.

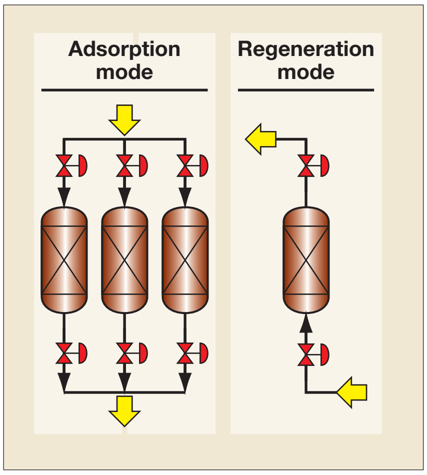


Figure 2 Schematic diagram of the dehydration unit studied

To perform such a process, water saturated natural gas from the upstream unit is sent to the molecular sieve dehydration plant where the gas stream passes through a separator to retain any free water carry-over from the upstream facilities. It is then routed to the molecular sieve dryers. A dehydration package consists of four dryers loaded with a special type of molecular sieve 4A; at any time three dryers are in adsorption and one in regeneration. The feed stream is split into three identical streams, each of which passes downward through one of the beds that are in adsorption mode (see **Figure 2**).

Dry gas streams leaving the adsorption beds are joined and passed through a filter to retain any solid particles coming from the dryers. Finally, dry and filtered gas is sent to the municipal gas station via a transmission pipeline.

Each adsorption cycle takes eight hours. After that, the dryer is switched to regeneration mode for removing the residual water. At once, that bed which has completed the regeneration step is replaced. During the regeneration process, a regenerative gas stream is passed through a heater where it is heated to approximately 270°C. This hot gas passes upwards through the offline saturated dryer heating the molecular sieves. As the sieves are heated up, adsorbed water begins to desorb and is carried away by the hot gas. The operating conditions of the target adsorption and regeneration processes and specifications of their feeds are shown in **Table 1** and **Table 2**, respectively.

Adsorption and regeneration operating conditions

Specifications	Value
Adsorption temperature, °C	47
Adsorption pressure, kPa	9101
Adsorption mass flow, kg/h	2.409e+05
Regeneration temperature, °C	270
Regeneration pressure, kPa	7929
Regeneration mass flow, kg/h	4.751e+04

Table 1

Mathematical modelling of regeneration

A computational fluid dynamic modelling technique was used to model the momentum, heat content and mass transfer of fluid through porous media, and also to investigate the refluxing phenomenon in the regeneration process studied. To solve these set of equations, commercial software (Comsol Multiphysics Ver. 4.2) was employed that utilises the finite element method to discretise partial differential equations to ordinary differential equations and finally solve them. The following assumptions are considered during the mathematical procedure:

- To reduce computation time, 2D axisymmetric mode is assumed
- The gaseous phase is an ideal gas
- Entrance and exit effects are negligible
- There is no slip condition near the dryer wall.

Governing equations

Mathematical modelling of the target regeneration process is obtained by coupling a set of general equations (including continuity, momentum, energy and mass balances), and particular equations such as physical properties, adsorption and desorption isotherms and equation of state as follows:

Continuity equation:

$$\frac{\partial \rho}{\partial t} + \nabla \cdot (\rho u) = Q_{br}$$

Momentum equation:

$$\frac{\rho}{\varepsilon_p}\left(\frac{\partial u}{\partial t} + (u \cdot \nabla)\frac{u}{\varepsilon_p}\right) = \nabla \cdot \left[-PI + \frac{\mu}{\varepsilon_p}(\nabla u + (\nabla u)^T) - \frac{2\mu}{3\varepsilon_p}(\nabla \cdot u)I\right] - \left(\frac{\mu}{k_{br}} + \beta_f |u| + Q_{br}\right)u + F$$

Energy equation:

$$(\rho C_p)_{eq}\frac{\partial T}{\partial t} + \rho C_p u \cdot \nabla T = \nabla \cdot (k_{eq} \nabla T) + Q$$

Mass equation:

$$\frac{\partial c_i}{\partial t} + \nabla.(-D_i \nabla c_i) + u \cdot \nabla c_i = R_i$$

In these equations, ρ (kg/m^3) is the density of the fluid; t (s) is the time; u (m/s) is the velocity vector; Q_{br} (kg/m^3·s) is the mass source or mass sink; εp is the porosity of bed; P (Pa) is the pressure; μ (kg/m·s) is the dynamic viscosity of the fluid; κ (m^2) is the permeability tensor of the porous medium; βF (kg/m^4) is Forchheimer drag option; F (kg/m^2·s^2) is the influence of gravity and other volume forces; $(\rho Cp)_{eq}$ is the equivalent volumetric heat capacity at constant pressure; T (K) is the bed temperature; C_p is the fluid heat capacity at constant pressure; k_{eq} is the equivalent thermal conductivity (a scalar or a tensor if the thermal conductivity is anisotropic); Q is the heat source (or sink); c is the concentration of the species (mol/m^3); D is the diffusion coefficient (m^2/s), and R is the reaction rate expression for the species (mol/m^3·s). Furthermore, the major particular equations are the Langmuir adsorption isotherm and ideal gas law. The proposed equations in 2D axisymmetric mode have been solved using the required initial and boundary conditions.

Feed and regeneration gas compositions

Components	Adsorption	Regeneration
Methane, wt%	72.95	73.1
Ethane, wt%	8.13	8.14
Propane, wt%	4.1	4.11
i-Butane, wt%	1.22	1.22
n-Butane, wt%	1.56	1.56
i-Pentane, wt%	0.00	0.00
n-Pentane, wt%	0.140	0.141
n-Hexane, wt%	1.73	1.73
n-Heptane, wt%	0.1897	0.19
n-Octane, wt%	0.1622	0.1625
n-Nonane, wt%	0.0337	0.0338
CO_2, wt%	3.79	3.8
Nitrogen, wt%	4.57	4.58
H_2O, wt%	0.1573	0.000

Table 2

SULZER

Sulzer Chemtech

Tower Technical Bulletin

Design and Installation of Cartridge Trays

Introduction

Cartridge trays, also known as package trays, are generally used for tower diameters in the range from 12” (300mm) up to 36” (900mm). For tower diameters below 36”, the installation of segmental trays is difficult and packing is often preferable over trays. Ultimately, the decision on what technology to use comes down to process requirements and economics.

As can be seen below, the design and construction of cartridge trays is unique and a bit complex. Cartridge trays typically consist of one or more bundles of 6 to 10 trays stacked together and connected with several tie rods running through the bundle. This can be a challenge; the trays must be assembled with near perfect alignment to ensure trouble-free installation. The resistance of the tray seal rings increases the force required to install and remove the tray bundles so proper design and correct dimensions are critical. Alternatively, Sulzer also offers Slit Trays^{TM} for smaller column diameters. These trays are installed individually to help minimize installation issues.

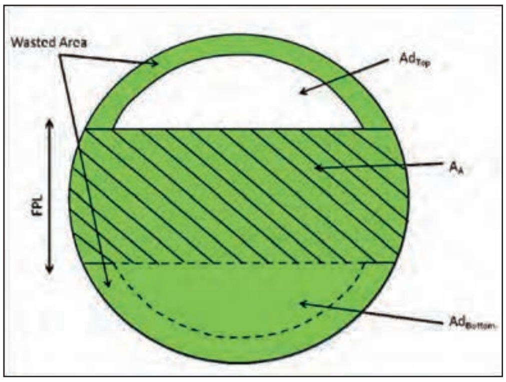
Bundles of Cartridge Trays

Design Considerations

The unique aspects of cartridge tray designs are both mechanical and process related. The trays must have a perimeter deck seal that maintains its integrity while the bundle is installed. The downcomers, which cannot seal to the column wall, must use an envelope design which results in some wasted area behind the downcomer (shown on the sketch to the right). In order to properly rate these trays hydraulically, the wasted area must be accounted for to ensure that it is not inadvertently counted as active area, AA.

The trays must be fixed together in a bundle form along with a mechanism to support the bundle within the column. The trays should be partitioned to maintain a maximum bundle length of 13ft (4m) for ease of handling.

The mechanical design of the cartridge trays should also be stronger than segmented trays since as they will be transported in assembled condition. The tray thickness should be increased (e.g 12ga or 2.5mm when referring to stainless steel) to maintain rigidity and ensure a tight fit. Stronger tie-rods and Schedule 80 spacer pipes should be specified as well. Since the gaskets are more prone to distortion, it is preferable to install them after the trays arrive on site. The selection of gasket material should be based on temperature and service. Metal gaskets of a suitable material are often preferred for their mechanical durability.



Cartridge Tray Cross-Sectional Layout

Important Tips

During installation, the orientation of the trays with respect to nozzles should be fixed prior to the bundle insertion as it will be more difficult to rotate afterwards. Also, access around the outside of the column must be properly allocated during the design process to ensure that there is no external interference with the bundle during insertion or removal.

Standard pipe sizes are typically used for columns with cartridge trays. Care must be taken during construction not to compromise the diameter and roundness of the column to ensure that the tray bundles will pass through the without interference.

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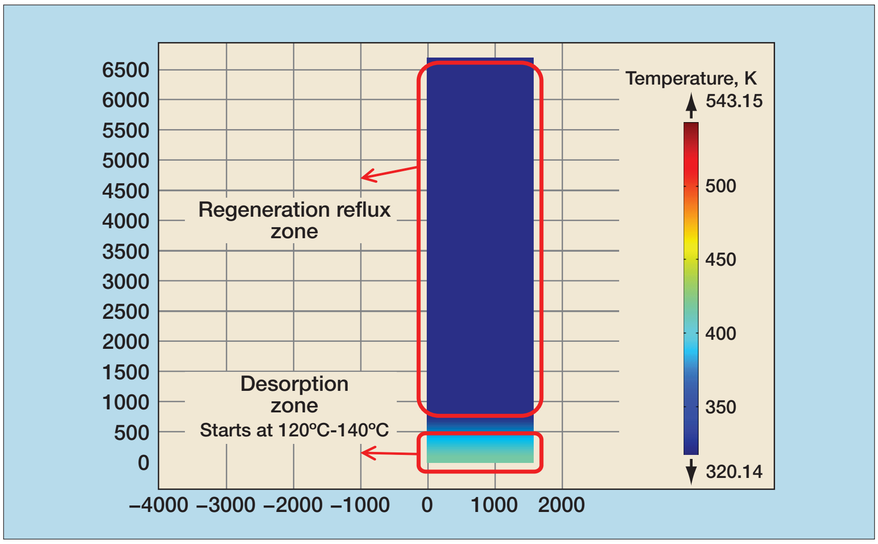

Figure 3 Temperature distribution during the regeneration process

Results and discussions

Figure 3 shows the temperature distribution of the adsorption bed at an early stage in the regeneration process. As is apparent in this figure, a high regeneration gas temperature (without enough ramp-up) leads to a large temperature gradient along the bed, and creates reflux at the early stages of the regeneration cycle.

At these operating conditions, due to the high pressure of the regeneration gas, high moisture concentration and a large temperature gradient are inevitable. For the design case, the licensor charged a molecular sieve with enough strength against reflux which could work more than four years without any malfunction. But for the next loading, a regular molecular sieve, manufactured by another company, could not withstand those conditions. It was observed that, only three months from the start of run, the loaded molecular sieve was ruined due to the reflux phenomenon. It also increased the pressure drop of the dryers. Therefore, it can be concluded that the molecular sieve, especially the binder and additives, should be made of appropriate raw materials to be capable of resisting the reflux phenomenon and preventing operational malfunctions.

As **Figure 3** shows, for our case study liquid water moved downward until it encountered the heating zone. At this point, boiling water created a reflux which ground the molecular sieve into a powder. Since certain components of the binder were somewhat soluble in boiling water, the molecular sieve subsequently became a wet cake (mud) which was then baked by the rising hot gas. These soluble components could ion exchange with the zeolite and/or combine with anions in water to form solid salts (Na_2CO_3, $CaCO_3$, $MgCO_3$, $NaNO_3$, and so on). These solid salts could then paste the remaining pellets or beads together to form a solid mass. This solid mass, formed in an annulus shape with a centre opening of less than one foot, did not allow gas to pass through, and consequently reduced the effective diameter of the bed (see **Figure 1**).

A high regeneration gas temperature (without enough ramp-up) leads to a large temperature gradient along the bed

Therefore, boiling water destroyed the molecular sieve such that the severity of the operating conditions should be greatly reduced to extend the replacement period of the adsorbent. The regeneration reflux showed some undesirable effects on the adsorption process which can be summarised as follows:

- Molecular sieve particle break-up
- Increasing pressure drop
- Gas channelling
- Premature water breakthrough

which all lead to poor adsorber performance.

As a consequence, these effects increased the reflux phenomenon with the following malfunctions:

- High pressure regeneration gas
- High moisture concentrations
- Large temperature gradients
- High degree of solubility of

Recommendations and consequences to prevent reflux phenomena

Recommendation	Consequence
1 Decreasing the regeneration gas pressure	• Needs compressor • Higher operating cost
2 Regeneration gas temperature ramp-up	• Hot oil system modification (if applicable) • Higher regeneration cycle time • Adsorption cycle time limitation
3 Layer of activated alumina at the top of the bed	• This approach may minimise the rolling boil but cannot fix the problem. Based on Figure 3, the reflux happens through the bed because of a high temperature gradient, so it can only reduce the reflux. We can consider it a modification.
4 Change the heating gas flow direction from the top to the bottom of the bed	• This is costly. Co-current regeneration requires more gas for stripping the bed completely. • The downward flow pushes heavy liquid contaminants, and possibly increases fouling rate.
5 Try to reduce the heat loss through the top of the bed by adding extra insulation and even installing a steam tracer	• This can only reduce temperature gradient between the top and bottom of the vessel.
6 Reverse all flows	• Bed fluidisation (lifting)
7 Using a special molecular sieve	• The bed can possibly operate without any problem.

Table 3

binder materials in water

- Choosing an inappropriate flow direction in adsorption and regeneration.

Recommendations and consequences

The recommendations proposed in **Table 3** can decrease the reflux phenomena which are reviewed in brief for the target gas dehydration unit.

According to recommendation 7 in **Table 3**, a special molecular sieve 4A (with high resistance against reflux phenomena), manufactured by Shanghai Hengye Chemical Co., was loaded into the target dryers about one year ago. To date, the dehydration unit has shown a good performance and no malfunction has been observed.

Acknowledgment

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To date, the dehydration unit has shown a good performance and no malfunction has been observed

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