Extending the life of molecular sieve beds

In this article **V K Arora** of Kinetics Process Improvements, Inc. presents the key lessons learned to improve the life of molecular sieve beds in two large ammonia plants that were failing after less than half the expected life span of 60+ months, mainly due to excessive pressure drop build up. A holistic approach greatly helped in identifying the root causes. A similar approach can be applied in molecular sieve systems in most other industries to improve plant reliability.

Molecular sieve drying systems were added in two large ammonia plants as part of a revamp to provide the final synthesis gas purification by removing water of saturation, carbon dioxide and ammonia. Since the commissioning, the molecular sieve beds in both plants lasted significantly shorter run times compared to the typical expected life in excess of 60 months. The shorter life was due mainly to an excessive pressure drop build up over time.

The molecular sieve beds were configured in an up-flow mode with internal refractory lining and typical steps for absorption, regeneration and standby times. Each bed was loaded with type 10 x 20 molecular sieves.

The up-flow configuration is not the usual design for molecular sieve systems in ammonia plants and initially this was considered to be the main reason of the problem by the suppliers.

Following the premature replacement of four charges of molecular sieve beds, a detailed study was commissioned to review and analyse all possible causes of the short life span. A holistic approach of the complete system identified several other relevant and cost effective solutions besides a detailed review of the up-flow configuration.

Observations during inspection

The following key observations were made during the inspection of molecular sieve vessels before the change out of beds:

- caking of the bottom few feet of the beds;
- damage of the top and bottom insulation screens;
- pulverisation of the desiccant at the top of the beds.

Study methodology

Initially, the customer worked through the recommendations of the various molecular sieve suppliers but the problem persisted with two consecutive charges of molecular sieves. Suppliers mainly insisted on reversing the flow as the solution to the problem. At this point, the customer engaged KPI to provide an independent review and recommendations before implementing any of the changes.

KPI followed a holistic approach in reviewing the complete system design within and around the molecular sieve beds along with the inspection reports and the operating history to identify all the potential causes.

To support a thorough analysis of the system, KPI independently developed suitable models to verify the existing design of molecular sieve beds and the complete system with further review of alternative practical options.

The model provided the estimates of minimum fluidisation velocities, pressure drops, channelling, regeneration heating/cooling time and flow requirements using different sizes and types of molecular sieves with different volumes of mono/composite bed combinations and different regeneration gas compositions.

After verifying the existing design and related issues through the model, various alternative combinations were simulated to check if the problem could be mitigated without any changes to the current flow configuration.

During the study, KPI also gathered relevant inputs from molecular suppliers to further verify the results before putting together the recommendations.

Study findings

Based on the initial review and evaluation, the study findings indicated a combination of factors contributing to an excessive high pressure drop build up in the molecular sieve beds resulting in shorter life. The key contributing factors identified are listed below:

- incipient fluidisation during adsorption in the up flow mode
- upstream separator limitations resulting in liquid carry over
- under regeneration
- freezing/thawing of bed
- mal-distribution and bed channelling

Incipient fluidisation during adsorption in the up flow mode could potentially result in particle breakage and dusting of molecular sieves, particularly in the top portion of the bed which could contribute to pressure drop build up. KPI estimated as much as 83% of the minimum fluidisation velocity, which was a sufficient cause of concern as a contributor to the higher pressure drop.

Caking of the molecular sieve bed in the lower few feet was observed in most of the beds. Liquid carry over was suspected. The lower part of the bed being the wettest and the weakest will be prone to the most damage to sieves especially with potential liquid carry over and contaminants likely to cause agglomeration into clumps as observed in the beds. Further, the smaller particle size of type 13X sieve does not provide the needed robustness and stability in the bottom of the bed for an up flow configuration.

A review of the upstream separator design indicated the following limitations in the upstream separator;

- undersized feed and vapour nozzles;
- inadequate demister pad.

The upstream separator is located upstream of the molecular sieve vessels.

The estimated high velocity through the feed nozzle at the operating loads with direct impingement on the existing baffle could also create a droplet shatter generating significant entrainment of much smaller droplets (< 10 microns) and uneven distribution leading to liquid carry over, which will have a detrimental effect on the sieves as these droplets will likely act as a hammer in the bottom sieves resulting in breakage, higher pressure drop and potential agglomeration due to over-saturation. The liquid carry over could also be a potential source of contamination of sieves further affecting its life.

The existing separation device and internals in the upstream separator were considered inadequate to efficiently capture the smaller droplets in the single digit micron size range.

Based on the bed configuration and operating loads and regeneration flow rate and temperature, the seven hours heating time was estimated to be grossly insufficient. KPI estimated about 14 hours heating time was needed for adequate regeneration, which was further verified through vendors.

From the operating data, it was noticed that the bed temperatures tend to fall below the freezing point of water to as low as 26°F (-3.3°C) after depressurisation. Even though sieves are suitable to handle such low temperature but the water freezing and subsequent thawing/revaporisation by quick heating in the molecular sieve pores may potentially result in breakage of the particles adding to the pressure drop problems.

The following additional concerns were listed for potential mal-distribution and channelling as contributors to pressure drop build up:

- absence of ceramic balls either on top or bottom of the bed – the absence of ceramic balls will result in an uneven flow distribution besides the potential plugging of the bottom screen which will not only worsen the flow distribution but will also lead to screen damage due to the resulting higher differential pressure of channelling;
- liquid carry over coupled with particle attrition/breakage will further worsen the flow distribution leading to bed channelling and higher pressure drop.

Mitigation options

Mitigation options were considered for the molecular sieve beds, upstream separator and flow configuration.

Molecular sieve beds

- Increase the regeneration time.
- Slow down ramping of temperature to minimise thermal gradient and potential spalling of sieves with quick vaporisation.
- Replace the 13X pellets with 4A type beads to reduce the incipient fluidisation and to provide robustness and stability of operation
- To avoid bed temperature going below freezing and subsequent thawing, marginally increase the feed temperature by a few degrees, subject to syngas compressor limitations, if any, and slow down the depressurisation rate.
- Install ceramic balls with floating mesh and a hold down grid to reduce the effect of incipient fluidisation
- Consider using a composite bed to mitigate the shortcomings of the up flow design (for adsorption), e.g.use a composite bed-1/8" at the bottom (75% vol) and balance 1/16" at the top (25%) with the advantage of minimising the incipient fluidisation and lower pressure drop through the bed. However, a larger regeneration flow along with a larger regeneration heater with more steam consumption may not favour this option

Upstream separator

Modify the suction drum while retaining the vessel and its nozzles:

- install an even-flow feed distributor to mitigate the effect of undersized feed nozzles;
- replace the existing demister pad with a higher efficiency demister or a combination of vane and demister pad to capture smaller droplets.

A properly functioning upstream separator provides the least expensive insurance for sustained performance of the molecular sieve system.

Change flow configuration

To avoid the addition of a regen exchanger and additional steam and regen flow as noted with the composite bed option, reversing the flows (meaning adsorption and cooling down flow and heating up flow) remains another option. This option will also require changes to piping, instrument settings, control logic and its testing along with a Hazop which needs to be carefully reviewed and engineered. This may also require a longer down time.

Recommendations

As a result of the detailed study and practical considerations, the following recommendations were made and carried out in both ammonia plants:

- no changes in the up-flow configuration are warranted;
- modify the upstream separator internals with an even flow distributor and an efficient demister;
- provide heat tracing at the inlet of the molecular sieve beds;
- increase the regeneration heating time;
- marginally increase the feed inlet temperature to molecular sieve beds;
- replace the existing type 13X molecular sieve with type 4A molecular sieves;
- install ceramic balls in the top and bottom of the beds along with floating mesh screens and a hold down grid;
- reduce molecular sieve bed volume.

Implementation and performance

Following the review of the study, the plant decided to implement the recommendations along with a change of internals of the upstream separator in both ammonia plants.

KPI carried out the engineering and supplies of the recommended internals as custom designed. Engineering including custom design/fabrication were completed on an urgent basis within three months to meet the turnaround schedule of the plant. They were successfully installed and fitted without any issues.

Following the implementation of the recommended changes, all four molecular beds have been performing well for nearly 40 months on the same charges. Based on the lower pressure drop trend since the operation of the modified system, the beds are expected to last for many more years before their replacement.

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