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Technical Guideline

Drying of Adsorbents

The following guideline applies to PuriStar® R3-12, PuriStar® R3-15 and R3-16 (in oxidic form), R3-22, R3-26, R9-PAR, R9-SR, R9-12, Selexsorb® AS, AS5 and E 315.

Introduction

PuriStar® R3-12, R3-15, R3-16, R3-22, R3-26, R9-PAR, R9-SR, R9-12, Selexsorb® AS, AS5 and E 315 are used in the oxidized state without any further pre-treatment step such as a reduction. The adsorbents as produced contain a few wt-% of adsorbed water, and they may pick up additional water from the atmosphere during handling and loading. If the material is not dried out before being brought on-stream, the adsorbed water will be desorbed into the process stream passing through the adsorber vessel during the first few days of operation. The concentration of water in the vessel effluent during this period will vary as a function of space velocity, temperature, time onstream and the nature of the process stream.

With liquid propylene, the water content coming out of the bed may start out as high as 1,000 – 2,000 ppm-wt. The water content will gradually drop as the bed dries out, but it could take several days before the product becomes in-spec on water. If there is no dryer downstream, it may be preferable to dry the catalyst bed before bringing it on-stream.

Drying can be accomplished by passing a dry, non-reactive gas (e.g. nitrogen) through the bed at temperatures of 180°C (360°F), preferably at a space velocity of at least 300 hr^{-1} (GHSV) and with the flow streaming down through the bed to ensure efficient and uniform drying. The following procedure describes this in more detail.

Temperature should be limited to 220°C (430°F), to avoid thermal stress on the adsorbent. In case this temperature is exceeded, shut down the heating but continue with the gas flow.

Alternatively, the adsorbent can also be dried on-stream. Some indications on doing this are provided after the drying procedure with a dedicated gas stream.

If the process stream contains H_2 (starting around 2 % vol/vol), the bed should be reduced prior to putting it onstream. This applies to all materials except PuriStar® R9-PAR and R9-SR, R9-12 and E 315 which are typically stable in H_2 containing streams. For further clarifications do not hesitate to contact BASF for further information.

A separate drying step as described in the present guideline is not required if the material is to be reduced.

Drying Procedure

Drying can be described by the following equation:

$$H_2O_{adsorbed} \rightarrow H_2O_{vap}$$

with ΔH_{vap} = 44,2 kJ/mol

The indicated value applies to liquid water but can also be used as an approximation for adsorbed water.

Drying can be accomplished by passing heated gas through the adsorbent bed. Down-flow is preferred for the drying step, as this promotes faster drying. Indeed, in down-flow the force of gravity helps remove any water droplets that could form downstream of the temperature front as the gas moves through the bed (in up-flow, gravity could limit the entrainment of water droplets).

Although Nitrogen is generally used for the drying, another gas such as methane, ethane or propane can be used, provided that the gas is dry, does not react with any of the indicated adsorbents, and is free of other species that would react with adsorbents (e.g. arsine, H_2S, COS, mercaptans, H_2). Please review the relevant product data sheet or contact BASF for further information.

The gas flow rate should be at least 300 Nm^3/hr per m^3 of adsorbent (i.e. a space velocity of at least 300 h^{-1}) to promote uniform and effective drying. A higher flow rate will decrease the time required to dry the bed (even a 5 x higher flow would typically be in down-flow operation no problem for the bed). For commercial size treaters, however, the gas delivery capacity may dictate drying at space velocities closer to the minimum 300 h^{-1}.

The gas temperature initially should be no higher than 50°C (120°F) to avoid thermal stress to the adsorbent. The gas temperature should be increased at a rate not in excess of 50° C (90°F) per hour, until the gas temperature reaches 200°C (390°F) or the maximum temperature possible with the gas supply system (whichever is lower).

Maximum gas flow at the maximum temperature should be maintained until the dew point at the exit of the bed stabilizes indicating that the bed is dry. A portable dew point analyzer can be hooked up for the determination of the dew point.

This procedure usually takes 1-2 days, depending on the actual gas flow rate and temperature and the initial moisture content of the bed.

Drying On-stream

In certain cases, the stream, which is to be treated with the freshly loaded BASF adsorbent, is also subjected to a drying step (e.g. with a mol sieve or alumina). In these cases, it can be evaluated on whether the BASF adsorbent can be dried with feed or product (on-stream) instead of doing a dedicated drying step, while at the same time avoiding any contamination with water downstream. To be able to do an on-stream drying, the following minimum conditions need to be fulfilled:

- The stream passing the freshly loaded BASF adsorbent as well as a drying adsorbent can be recycled to the inlet of the BASF adsorbent bed
- The drier to be used to treat the stream coming from the adsorber loaded with the BASF adsorbent has sufficient capacity to handle the initial higher loads of water desorbed from the BASF adsorbent.

If both conditions can be fulfilled, a drying on-stream can be considered. The drying on-stream consists then simply of sending the dry feed/product stream over the freshly loaded BASF adsorbent, drying the stream coming from the adsorber and recirculating this stream to the inlet of the adsorber.

Important Note! If the adsorbent has been dried and the process stream will contain olefins, the bed must be cooled as uniformly as possible to below 50°C (120°F), preferably at a space velocity of at least 300 hr^{-1} and a cooling rate not in excess of 50°C/hr, before going on-stream.

This measure is necessary to ensure that no hot spots remain in the bed. Hot spots could result in an exothermic oligomerization or decomposition of olefins, which could coke and incapacitate the adsorbent.

About Us

BASF is a leading global manufacturer of catalysts for the chemical industry, with solutions across the chemical value chain. The business comprises chemical catalysts, adsorbents and custom catalysts. Priority is given to developing new and improved products that enable the chemical industry transformation to net-zero emissions.

BASF's chemical catalysts and adsorbents business is part of the company's Performance Chemicals division. The division's portfolio also includes refinery catalysts, fuel and lubricant solutions, as well as oilfield chemicals and mining solutions. Customers from a variety of industries including Chemicals, Plastics, Consumer Goods, Energy & Resources and Automotive & Transportation benefit from our innovative solutions.

BASF - We create chemistry

Americas
BASF Corporation
Phone: +1-732-205-5000
Email: catalysts-americas@basf.com

Asia Pacific
BASF (China) Company Limited
Tel: +86-21-2039 2549
Phone: catalysts-asia@basf.com

Europe, Middle East, Africa
BASF Services Europe GmbH
Phone: +49-30-2005 5000
Email: catalysts-europe@basf.com

www.chemical-catalysts-and-adsorbents.basf.com

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