

Drying of Adsorbents

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

Introduction

PuriStar® R3-12, R3-16, R3-22, R9-PAR, R9-SR and Selexsorb® AS 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 drawn out by the process during the first few days of operation. The concentration of water in the reactor 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, nonreactive 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.

If the process stream contains more than 2 vol-%

H₂, the bed should be reduced prior to putting it onstream. This applies to all materials except PuriStar R9-PAR and R9-SR, which are not used in the reduced state. Drying is not required if the material is to be reduced.

Drying Procedure

Drying can be accomplished by passing heated gas through the bed. Down-flow is recommended for the drying step, as this promotes faster drying. Indeed, in down-flow the force of gravity helps remove water droplets that can condense as the gas moves through the bed (in upflow, gravity is working to keep the water droplets in the bed).

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, H₂S, COS, mercaptans, H₂). Please review the relevant product data sheet or contact BASF for further information.

The gas flow rate should be at least 300 Nm³/hr per m³ 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 be 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 catalyst. 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 this measurement. This procedure usually takes 1-2 days, depending on the actual gas flow rate and temperature and the initial moisture content of the bed.

Important Note! If the adsorbent has been dried and the process will contain olefins, the bed must be cooled 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 decomposition of olefins, which could coke up and incapacitate the adsorbent.

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