

Selexsorb[®] CD/CDX/CDL/CDN

Adsorbents for the removal of polar compounds

The present guideline provides information on how to bring the indicated adsorbents into contact with liquids and gases.

Background

Selexsorb CD/CDX/CDL/CDN are often used for the treatment of hydrocarbon streams in liquid or vapor phase operation. These hydrocarbon streams can contain smaller or larger amounts of olefins or even multiple unsaturated components (like acetylenes). All these unsaturated components can either react with themselves or with each other if heated to a sufficiently high temperature.

High temperatures can occur if beds are not properly cooled down after regeneration or when brought first into contact with the respective hydrocarbon streams. Certain hydrocarbons can indeed exhibit high energies of adsorption, which, if not properly addressed, can lead to local temperature increases and therefore to reactions of unsaturated components.

Selexsorb CD/CDX/CDL/CDN have a very long history of being started up with no or only minor temperature increases observed. Selexsorb materials have been specifically developed for allowing start-up with olefins present, avoiding the typical pre-loading procedure as e.g. used for certain mol sieves like 13X.

In the following, measures are indicated to minimize local temperature increases.

Before any of the fillings described in the following is carried out, it is imperative to check whether at all enough volume of the gas or liquid is available to fill the vessel within the indicated time frame. The required volume needed can be approximated with

the volume of the empty vessel minus 1/3 of the volume filled with adsorbent.

Contacting with Gases

The following indications are based on the example of a stream consisting mostly of ethylene.

Typically, the vessel is kept under an inert gas (nitrogen, fuel gas free of olefins or other). The adsorbent must be properly cooled down (a regeneration is typically done before bringing the adsorbent in contact with any stream to be treated).

- If nitrogen is available with sufficiently high pressure, the vessel can be pressurized to operating pressure with nitrogen alone and can then be lined out with the rest of the system. A continuous flow is then established through the vessel.
- If only lower pressure nitrogen is available (e.g. 8 bar (g)/120 psi (g)) the vessel can first be pressurized with nitrogen and then with feed itself up to the required operating pressure. However, this pressurization step with feed should be done as fast as possible (within a few minutes). Once the vessel is at operating pressure, the system is directly lined out and a continuous flow is immediately established through the vessel.
- Alternatively the vessel can also be pressurized with feed alone (again within a few minutes only) and must then be directly lined out with the rest of the system and a continuous flow through the vessel must be established.

Technical Guideline

In case any larger temperature increases are observed during pressurization, the procedure must be stopped, and the vessel must be directly depressurized. Depressurization is preferably done co-currently with the direction the vessel was being pressurized with.

Contact with Liquids

Before starting the filling with liquid, the following points must be checked in addition to points indicated earlier:

- The availability, flow rate and maximum discharge pressure of the pump used for filling the vessel. The pump should be able to provide for the volume to be filled in ½ hour at the starting pressure used in the vessel.
- The possibility to discharge sufficient gas/vapor during the filling of the vessel to maintain the pressure in the vessel. A starting point can be the volumetric flow rate assuming that the liquid to be filled is filled in ½ hour.

The following indications are based on the example of a stream consisting mostly of propylene (as example of an LPG type stream). Typically the vessel is kept under an inert gas (nitrogen, fuel gas free of olefins or other). The adsorbent must be properly cooled down to a temperature lower than 50°C / 120°F (a regeneration is typically done before bringing the adsorbent in contact with any stream to be treated). It should thus be avoided to start filling during the hottest part of the day. The additional difficulty with LPG type streams is that these can flash to very low temperatures (below the allowed lower temperature limits of the steel used for vessel and piping) if these liquids are flashed against too low a pressure.

- If nitrogen is available with sufficiently high pressure, the vessel can be pressurized to operating pressure with nitrogen alone. Once the vessel is at operating pressure, it will be filled from the bottom with the liquid feed while the nitrogen is vented. Once the vessel is

completely filled, the vessel will be lined out with the rest of the system and a continuous flow will be established without any delay. The filling with liquid must be done within 1 – 2 hours maximum.

- If only lower pressure nitrogen is available (e.g., 8 bar (g) /120 psi (g)) the vessel can first be pressurized with nitrogen and then with vaporized feed itself to the required operating pressure. However this pressurization step with the vapor should be done as fast as possible (within a few minutes). Once the vessel is at operating pressure, it will be filled from the bottom with the liquid feed while the gas phase (consisting of nitrogen and vaporized propylene) is vented. Once the vessel is completely filled, the vessel will be lined out with the rest of the system and a continuous flow will be established without any delay. The filling with liquid must be done within 1 – 2 hours maximum. Vapor for this type of operation might e.g. be coming from a propylene splitter. In this case it is preferable to use the propane rich vapor coming from the bottom of the splitter.
- A pressurization of the vessel with vaporized feed alone is not allowed. If pressurization with nitrogen is not possible, the adsorbent in the vessel must be pre-loaded. The pre-loading step often consists of sending the regeneration gas stream loaded with 1–2 vol % of vaporized feed through the vessel until the adsorbent is completely loaded with feed. This is of course only done after having properly cooled down the bed. Alternatively also nitrogen can be used as carrier gas. After the pre-loading has been completed, the vessel can be pressurized with vaporized feed alone to operating pressure.

In case any larger temperature increases are observed during pressurization, the procedure must be directly stopped and the system immediately depressurized preferably co-current to the direction of pressurization.

If during filling with liquid any larger temperature increases are observed, the procedure must be directly stopped, the vessel must be drained and depressurized and flushed immediately after with

an inert gas like nitrogen. Again, the depressurization and nitrogen purge steps are preferably done co-currently to the direction of pressurization.

Temperature increases of 20 – 50°C / 35 – 90°F have been observed depending on the filling rate applied. For estimating possible temperature increases use the heat of vaporization of the liquid being filled into the vessel as heat of adsorption.

The measures as indicated above in case of larger temperature increases during the pressurization or filling steps should be done if temperatures locally rise above 90 – 100°C / 190 – 210°F in the case of propylene and 100 – 120°C / 210 – 250°F for butenes and stay there for more than 10 – 20 minutes.

During fast depressurizations as indicated above, it is possible that the bed might have been locally disturbed. Please contact BASF for further review.

If in doubt about the right procedure to apply, please feel free to contact BASF.

Note

It must also be noted that all indications provided apply only to one vessel loaded with the respective Selexsorb adsorbent. Any procedure set up by the customer and trying to fill e.g. multiple vessel systems lined up in series or in parallel is the sole responsibility of the respective customer. Also, proper instrumentation must be in place to monitor the respective steps indicated above. Proper instrumentation are typically temperature measurements, pressure measurements and the case being the analysis of hydrocarbons in inert streams.

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