TS01.032016-H

Ultra Purification of pDCB

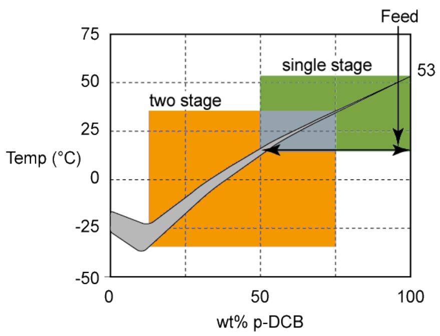
Suspension Crystallization with Wash Column Separation

Application

The growth in consumption of polyphenylene sulfide (PPS, an engineering plastic primarily used in electronic component manufacture) has fueled the demand for high-purity para-dichlorobenzene (pDCB). The smallest amount of impurities in the starting monomer can significantly affect the quality of the final product. Therefore, ultra-pure starting materials are essential. Crystallization provides the highest degree of separation and can guarantee production of ultra-pure pDCB isomer.

Crystallization is ideally suited to provide an efficient end-purification. The initial separation can be completed in a distillation column that provides around 90-99 wt% product. Suspension crystallization can then easily upgrade this to 99.95% with high recovery in a single step.

The phase diagram for a solution of DCB isomers is shown below. The feed concentration and the eutectic point determine the maximum recovery for any crystallization process.



Phase diagram of DCB isomers

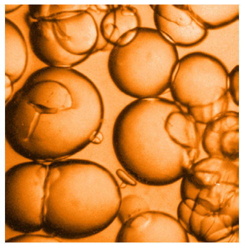
Suspension crystallization provides the maximum recovery over a single stage configuration due to the near ideal crystal growth conditions. Efficient separation of these ultra-pure product crystals insures the purity of the final product.

The temperature difference between the final product (pure melt) and the reject stream influence the separation efficiency. In many cases a single step is all that is required for high purity production at acceptable recoveries. One additional stage will provide the maximum recovery possible (by operating on or near the eutectic point) based on any feed concentration. The phase diagram for pDCB illustrates this relationship.

engineering for
a better world

gea.com

Ultra Purification of pDCB



Suspension Crystallization with Wash Column Separation

Features

- High product purity – The ultra-pure crystal from the suspension crystallization process and the efficient separation of the GEA wash column provides the maximum purity possible.
- High product recovery – The maximum product recovery is determined by the feed and eutectic compositions. Since p-DCB has a relatively low eutectic composition, high feed concentrations can provide recoveries over 97% in a single step.
- Continuous operation – The suspension crystallization process operates as a continuous process. Operating turndown ratio of 50% and an immediate stop/hold (for upstream/downstream upsets) does not require a complete start-up.
- Feedstock – Variations in feed composition are absorbed by the system. Expansion and debottlenecking – The suspension crystallization process is ideally suited to end-purification and can be easily added to existing units to improve capacity and final product purity.
- Economics – The cost of operation is strongly dependent on the specific requirements of the system. Due to the single crystallization step required by suspension based systems the operating costs are significantly lower than other separation techniques. GEA can provide assistance in determining the optimum configuration and cost information for your specific circumstances.

Process description

The process is based on crystallization in an industry proven scraped surface vessel crystallizer and final purification completed using the unique wash column separation technology. The crystallizer converts the feed into a crystal suspension of pure product crystals and the residual mother liquor. The GEA wash column separates this slurry into a pure product melt and the concentrated impurities as mother liquor.

The crystallizer consists of a jacketed vessel with a rotating scraper assembly. Refrigerant circulates in the outer jacket and cools the inner wall of the vessel.

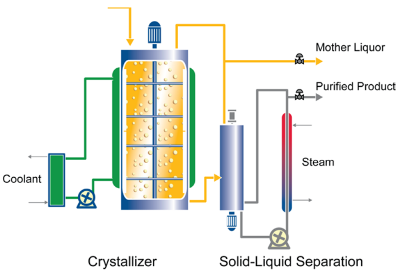
The scraper sweeps the wall surface and prevents build-up of crystals to maintain a clean heat transfer surface and continuous supply of product crystals.

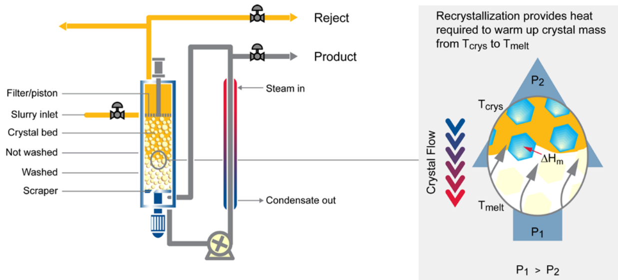
Each individual crystal provides growth surface that can absorb the supersaturation caused by cooling the product at the swept surface. With billions of individual crystals present, this will provide near ideal growth conditions and ensure the production of ultra-pure crystals. The resulting low growth rates possible in suspension based crystallization systems allows pure crystal production from even relatively impure mother liquor.

The GEA wash column completes the separation of this mixture of pure product crystals and residual mother liquor.

The crystal suspension enters the wash column assembly. A piston mechanically compresses the crystal suspension to remove the mother liquor and form a packed crystal bed. This bed consists of the pure product crystals surrounded by some residual mother liquor. The new crystals entering the column force the bed through the column toward the scraper assembly at the opposite end. The scraper disintegrates the crystal bed and a circulation pump provides melted product to reslurry the crystals. The circulation flow carries the crystals to a heat exchanger where e.g. steam or other heat sources provide the heat necessary to melt the crystals. The melted product leaves through a pressure control valve that provides the pressure needed to force the wash liquid through the packed crystal bed. The required pressure is adjusted depending on the level of the washfront. The washfront can be detected by the change in temperature between the washed and unwashed portions of the crystal bed.

The melted product in the recirculation stream countercurrently washes the residual mother liquor from the packed crystal bed as it moves through the column. The wash liquid forms an internal reflux loop and therefore does not need to be recovered as with centrifuge wash liquid. The crystal bed depth provides an extremely efficient wash zone for removal of the mother liquor ensuring complete removal of all impurities.





On-site demonstration of this technology is possible in various configurations using GEA pilot plants. For more information regarding this technology and your specific configuration requirements, please contact us or get in touch with your local GEA contact on gea.com via the Application Chemical, Specialty & Fine Chemicals.

GEA Netherlands

GEA Niro PT B.V.
De Beverspijken 7-b
5221 EE 's-Hertogenbosch, Netherlands

Tel +31 73 6390390
Fax +31 73 6312349

info.niropt.nl@gea.com
gea.com