TS02.032016-H

Purification of Organic Chemicals

Suspension Crystallization with Wash Column Separation

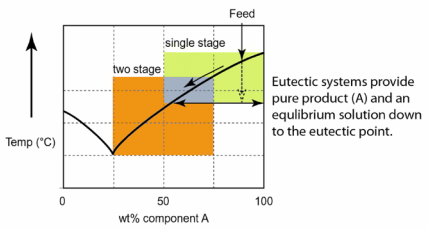
Application

The steady growth in the development of new products from a range of organic chemical intermediates has fueled the demand for high-purity starting materials. Existing processes also benefit from a critical study on the separation techniques used. Impurities can significantly affect the quality and economics of the final product making ultra-pure feedstock essential. Crystallization provides the highest degree of separation and generally guarantees production of ultra-pure products.

A wide range of organic compounds including the difficult separations presented by close boiling isomers, azeotropic solutions and enantiomers are highly suited for purification by crystallization. Over 80% of organic solutions are considered eutectic systems that provide the possibility for complete separation by crystallization in a single step. Many (>60%) of the known organic compounds have melting points below 100 °C with boiling points being much higher. Compounds that easily decompose or react at elevated temperatures are also highly suited for purification by crystallization.

The feed concentration and the eutectic point will determine the maximum recovery for any crystallization process. In general, crystallization is ideally suited to provide an efficient end-purification step, but the near ideal crystal growth conditions found in Suspension Crystallization provide pure crystals and acceptable recovery with a single stage. Efficient separation of these ultra-pure product crystals ensures the purity of the final product. In many cases only two stages are required for ultra-pure product and the maximum recovery possible (by operating on or near the eutectic point) based on any feed concentration.

The phase diagram for a typical organic solution illustrates this relationship.



Phase diagram of a typical organic solution

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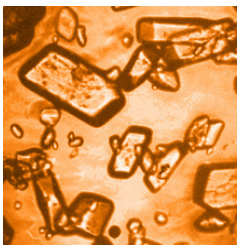


Features

- 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 for any crystallization system. Suspension crystallization provides the highest recoveries in a single step.
- Continuous operation- The suspension based crystallization process operates efficiently as a continuous process.
- Feedstock -Normal variations in feed impurity 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 continuous operation mode and 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.

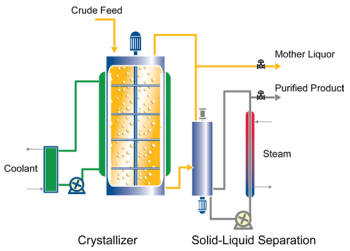
The scraper sweeps the wall surface and prevents build-up of crystals to maintain a clean heat transfer surface and continuous supply of product crystals.

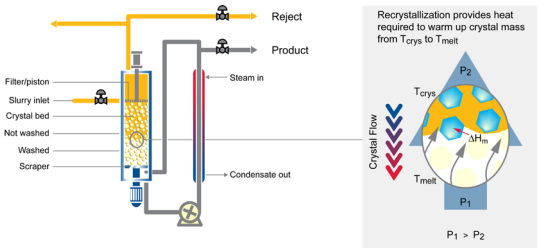
Each individual crystal provides growth surface that can absorb the supersaturation caused by cooling the product at the swept surface. With billions of individual crystals present, this will provide near ideal growth conditions and ensure the production of ultra-pure crystals. The resulting low growth 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.

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