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Ultra Purification of Hydrogen Peroxide

Suspension Crystallization with Wash Column Separation

Application

The GEA suspension based melt crystallization process with wash column separation exhibits some distinct advantages that favor its application over competitive technologies. Incentives to apply this process for highly explosive substances include:

- Purity - Very high purities (99.9+%) are typically attained in eutectic systems.
- Safety - The process is operated at the lowest possible operating temperatures assuring minimum thermal and chemical activity. The complete process is run in a closed, pressurized system without the presence of a vapour phase, thereby minimizing the risk of decomposition.
- Energy - It is a continuous process with the heat of crystallization being 1/3 to 1/7 of the heat of evaporation. Energy savings compared to layer crystallization are substantial. The utility consumption of a GEA process is typically between 2 to 10 times lower than the layer option, depending on how many stages are necessary for the latter technology. The savings result from the fact that the product is crystallized only once. Against that the layer process is commonly repeated a couple of times to achieve acceptable product purities. Also the heating up and cooling down of the complete steel of the installation adds up significantly to the energy consumption of a discontinuous layer process.

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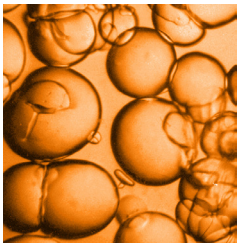


Example case

Together with the German Degussa AG, successful pilot plant test for the purification of hydrogen peroxide have been performed. Since hydrogen peroxide has a high energy content and decomposes to water and oxygen it is used as an oxidizer/propellant for aircraft rockets in space technology. Process safety has been the mayor incentive for Degussa when they decided to investigate the GEA technology for their application in more detail. At an impurity level in the crystallizer of 15 wt% purification ratios between 300 and 3000 have been achieved. During the tests hydrogen peroxide was produced at concentrations between 99.9 wt% and 100 wt% and at a surprisingly low amount of other impurities usually under or close to the detection limit of the individual components. The high efficiency of the wash column separation becomes apparent when comparing this with "normal" centrifuge separation. Wash column separation was typically a factor 10 better than conventional centrifuges.

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.

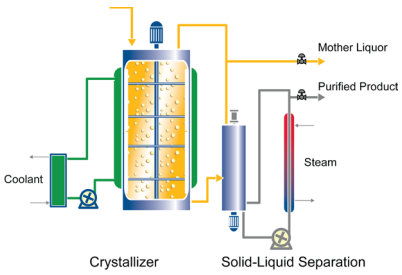
The scraper sweeps the wall surface and prevents build-up of crystals to maintain a clean heat transfer surface and continuous supply of product crystals.

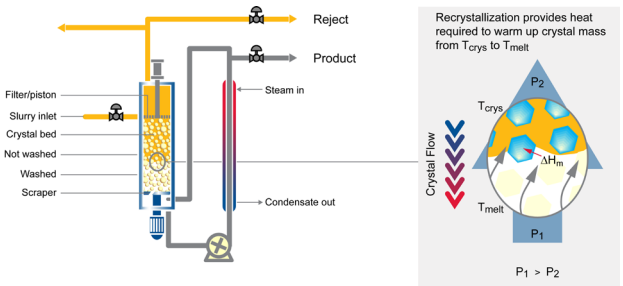
Each individual crystal provides growth surface that can absorb the supersaturation caused by cooling the product at the swept surface. With billions of individual crystals present, this will provide near ideal growth conditions and ensure the production of ultra-pure crystals. The resulting low growth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