

Ultra Purification of 4,4' MDI

Suspension Crystallization with the GEA Wash Column

Application

Methylene diisocyanate is used in the production of polyurethane plastics and foams. It is applied over a wide range of products including rigid plastics, paints and adhesives, automotive parts and foams for insulation, bedding and packaging.

The reaction process generates three main isomers; these are 2,4' MDI, 4,4' MDI and 2,6' MDI. The 2,4' MDI and 4,4' MDI are the most commercially important as their ratio in the starting product determines the stiffness of the foam produced when the isocyanate is reacted with a polyol. The monomers also react to form dimers that differ in solubility and produce a cloudy product.

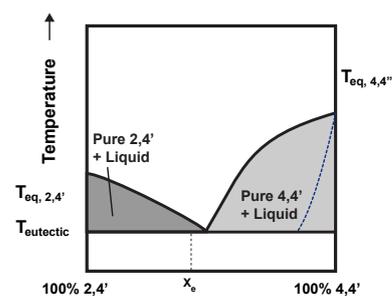
The phase diagram for a solution of 2,4' MDI' and 4,4' MDI' is shown below. The eutectic limits the separation of the isomers. However for MDI this composition is a desirable product for the further production of polyurethane foams.

Features

- **Flexible product purity** - Purity >99.5% down to the eutectic as needed.
- **Feedstock** - Operates on a range of feed stocks with the variations in feed impurity composition being absorbed by the system.
- **Economics** - Continuous operation and energy efficient suspension based crystallization ensure the operating costs are significantly lower than other separation techniques.

- **Maintenance** - Low rotating speeds and the robust construction of the GEA Purifier™ provide trouble free operation.

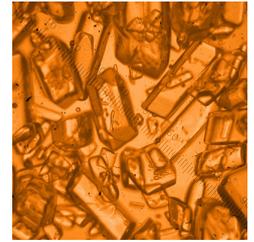
GEA can provide assistance in determining the optimum configuration and cost information for your specific circumstances.



Phase diagram of 2,4' MDI and 4,4' MDI

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Process description

The melt crystallization process is carried out in an industrially proven scraped surface crystallizer where final purification is completed in the GEA wash column. The feed product is cooled inside the crystallizer where part of the product is converted into pure crystal solids leaving the impurities concentrated in the residual mother liquor.

The crystallizer consists of a jacketed vessel with a rotating scraper assembly. Evaporating refrigerant or a secondary coolant in the outer jacket cools the inner wall of the vessel.

The scraper sweeps the wall surface and prevents buildup 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 GEA wash column completes the separation of this mixture of pure product crystals and residual mother liquor. This separation is based on GEA's patented wash column technology and is an essential component in this purification process.

The crystal slurry is compressed within the GEA wash column to remove most of 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 wash column will force the bed 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 any other heat source provides the heat necessary to melt

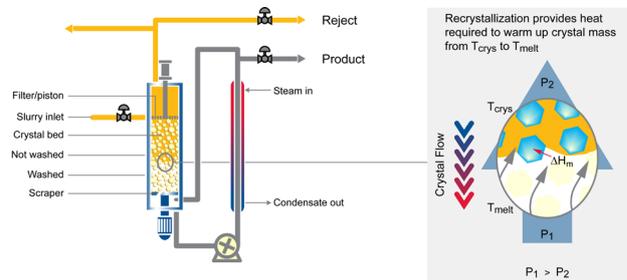
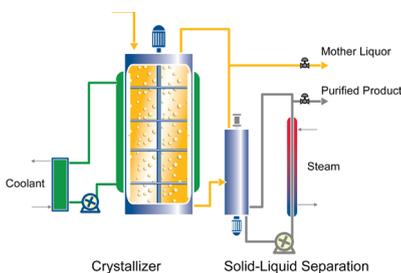
the crystals. The melted product can then be discharged to product storage.

The melted product in the recirculation stream countercurrently washes the residual mother liquor from the packed crystal bed as it moves through the wash column. The length of the crystal bed provides an extremely efficient wash zone for removal of any remaining impurities in the mother liquor ensuring ultra-pure product. Unlike other solid/liquid separation devices operating with a much shorter bed depth, the wash liquid forms an internal reflux loop and is therefore recovered as recrystallized product in the melt circuit instead of leaving with the mother liquor.

Experience

GEA is a technology oriented engineering company with over 40 years experience in the design and execution of crystallization plants worldwide. We started by crystallizing water from liquid food products in 1973 and since then have built and installed over 120 purification units around the world including applications for water, PX, PDCB, phosphoric acid as well as many other classified organic chemicals.

New developments in process technologies pose new challenges every day which require innovative and low-cost solutions. Practical orientation and customer requirement play a vital role in this connection. GEA consistently orientates itself towards the needs of the customer.



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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