MELT CRYSTALLIZATION

TNO innovation for life

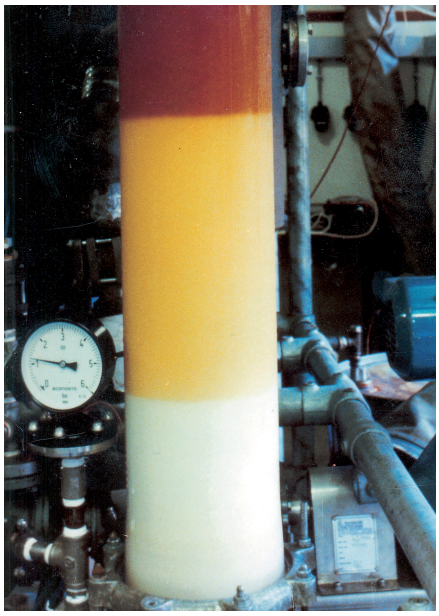
Melt crystallization is a valuable tool in your company for the production of ultra pure chemicals (well over 99.9% purity). TNO has developed the TNO HWC® for crystallization from the melt. This proprietary technology provides opportunities for producing ultra pure chemicals on a large scale with a low consumption of energy, without using solvents.

Main features of interest to the Chemical Industry

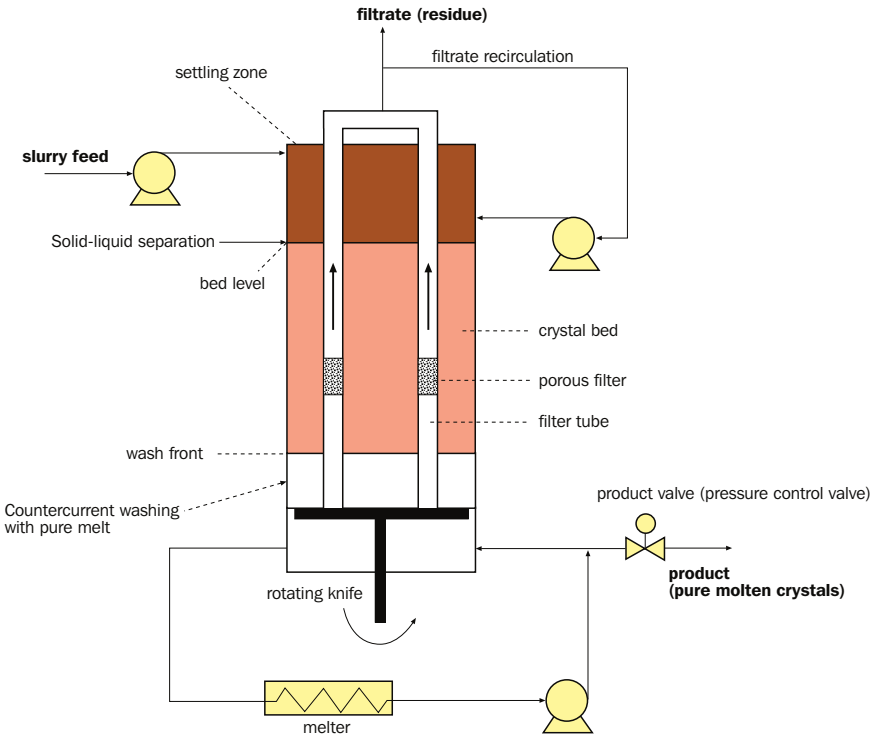
- Single step process usually combines high purity (> 99.9%) with high recovery.
- Low energy consumption.
- No solvent.
- Continuous operation.

THE MERITS OF MELT CRYSTALLIZATION

Melt crystallization differs from other crystallization methods in the fact that the operating temperature is close to the melting temperature of the main component. This enables the use of extra purification methods around the melting temperature, like washing with the pure melts, sweating, etc. In general terms, it may be said that melt crystallization's energy requirement, in principle, is low. The heat of transition in crystallization is typically two to five times lower than in distillation. Moreover, the need of reflux is limited due to the high purification efficiency. Melt crystallization is often superior on an economic level as well as from an environmental point of view for ultra purification and separation of isomers compared to other typical separation operations like distillation, adsorption



The TNO demonstration wash column operating with para-Xylene. The separation effect is visualized by addition of a dye to the mixture.



Schematic diagram of the TNO wash column

and absorption. Furthermore, the use of an organic solvent is avoided, which will have a positive impact on safety and the environment and on application in food industry. Due to the relatively low operating temperature compared with distillation, this process is often applicable.

TNO HYDRAULIC WASH COLUMN

When cooling a melt below the crystallization temperature in general relatively pure crystals will be formed, because the impurities with their deviating size and/or shape will not fit in the highly structured crystal lattice. In order to benefit from the high intrinsic purity of the crystals, these need to be separated almost completely from the impure mother liquor which contains most of the impurities. A TNO hydraulic wash column is perfectly suited for that task since it combines continuous solid – liquid separation with a highly efficient counter current washing operation. The wash liquid is usually a small part of the pure molten crystals and it is not consumed during washing (internal recycle).

Typically the wash column product will contain 100-1000 times less impurity molecules than the impure mother liquor from which the crystals were grown.

TNO wash column concept offers

- A straight forward scale up strategy.
- A broad turn down ratio.
- Control strategies for automatic operation.
- High specific throughput up to 15 ton/hr per m^2 cross sectional area.
- Low operating costs.

Survey of organic compounds of interest successfully investigated at TNO

Acetonitril	para-Chloronitrobenzene
Acrylic acid	para-Chloronitrobenzene
Caprolactam	para-Dichlorobenzene
Maleic anhydride	para-Xylene
Napthalene	Phenol

PROCESS DESCRIPTION

The TNO hydraulic wash column consists of a cylinder, in which a number of filter tubes of a constant diameter extend in axial direction. The wash column is fed with the suspension from a melt crystallizer. The mother liquor escapes via filters. The filters retain the crystals and a packed bed of crystals is formed within the cylinder. The bed is transported downwards by means of hydraulic pressure as the filtrate flows toward the filters. The bed is disintegrated at the bottom of the column and the crystals are suspended in the circulating melt and then tapped off as molten product. A part of the molten product is forced through the packed crystal bed and re-crystallizes on the colder crystals thereby producing the socalled wash front. The counter-current washing and the re-crystallization of wash liquid at the wash front cause a very good purification result. The three zones – feed suspension, unwashed and washed bed – are clearly visible in the photograph above.

Melt crystallization is especially interesting for low molecular weight organic feed mixtures (molecular weight < 200 g/mol) with a melting point between –50 and 200 °C. Next to 15 bulk scale applications up to 200 fine organic chemicals have been identified which can be purified with this technology.

Beside purification of organic chemicals, this technique is also suited for purification of inorganic materials (e.g. phosphoric acid, hydrogen peroxide) and metals (e.g. aluminium).

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