

Mass Transfer-I

Crystallization (Continue...)



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Crystallization (Continue...)

Crystallizers

CLASSIFICATION OF CRYSTALLIZERS

1) MIXED-SUSPENSION, MIXED PRODUCT REMOVAL (MSMPR) CRYSTALLIZERS

- These are the most common class of industrial crystallizers.
- It is also called circulating magna crystallizers.
- It circulates the growing crystals through the zone of the crystallizer where the supersaturation conditions are generated.
- This may be accompanied by mixed- or classified-product removal, and with or without destruction of fines.
- 20-40% solids in suspension is very common in these types of crystallizers.
- Two types of crystallizers are Forced Circulation Crystallizers and Draft-Tube-Baffle Crystallizers.

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Source: <https://www.chemengonline.com/a-clearer-view-of-crystallizers/?printmode=1>

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2) CIRCULATING LIQUOR/CLASSIFIED- SUSPENSION CRYSTALLIZERS.

- In this type, only the liquor or a weak slurry is circulated, while the bulk of the growing crystals are not circulated.
- Supersaturation is imparted to the liquor in one part of the equipment, whereupon this liquor is circulated to another area where it relieves the supersaturation on growing crystals.
- This type of crystallizer is also available with or without fines destruction capabilities. The units are usually identified as Krystal- or Oslo-type crystallizers.

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3) SCRAPED-SURFACE CRYSTALLIZERS

- Crystallization is induced by indirect heat exchange with a cooling medium at the heat-transfer surface, which is continuously scraped and agitated to minimize fouling.
- This type of equipment employs vertical tanks with scrapers or horizontal pipes.

4) TANK CRYSTALLIZERS

- Crystallization is produced by cooling the feed solution in either static or agitated tanks by natural convection and radiation, by surface cooling through coils or a jacket, programmed evaporative cooling, reaction or antisolvent methods.

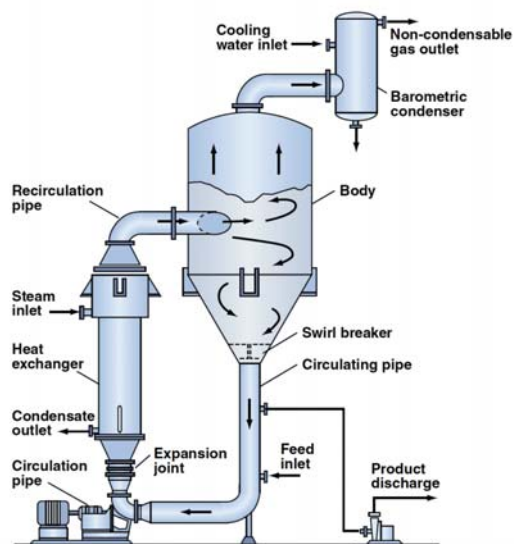
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Forced Circulation (FC)

- Advantage:
 - Can easily control circulation
 - rates and velocities
 - Low capital cost
- Disadvantages:
 - High heat
 - No stirrer \therefore large range of concentrations and temperatures
 - Full cross-section of vessel is not used for crystallization



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- The most widely used crystallizer is the mixed suspension mixed product removal MSMPR crystallizer or we can say the growing crystals are kept in suspension by agitation (almost 20 to 40 percent solids in the suspension)
- The most common one is the forced circulation or evaporative crystallizer.
- A circulating pump is installed to circulate the entire magma through the heat exchanger and the crystallizer.
- The slurry is pumped through an external steam heated vertical heat exchanger to raise its temperature ~ 2 to 6°C and no boiling occurs in the heat exchanger.
- The liquid velocity is maintained as ~ 2 to 3 m/s .
- The hot liquid is thrown into the crystallizer and the evaporation of the liquid occurs inside it.
- The evaporation of the liquid creates the required supersaturation for the crystallization.
- Many inorganic salts, such as, ammonium sulphate, sodium chloride, citric acid, sugar, etc. are crystallized in the forced circulation crystallizer.

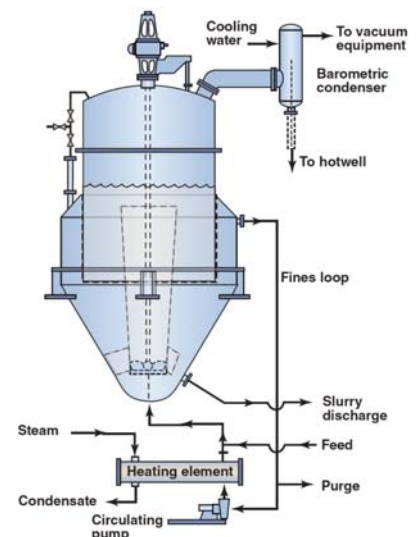
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Draft Tube Baffles (DTB)

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- Propeller inside fixed tube
- Preferential fines removal and classified product
- Little crushing of crystals
- Uniform concentration with little dead space
- Large crystals
- DTB is widely used in the industries
- The body of the draft tube crystallizer is provided with an inner draft tube and skirt baffle.
- A long shaft slow moving impeller throws the liquid upward
- The magma is fed from the bottom of the crystallizer.



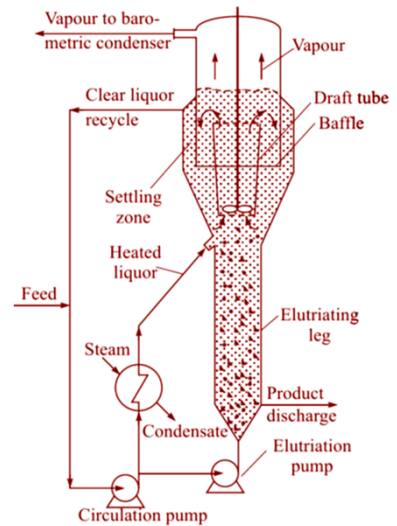
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- The bottom narrow part is called Elutriating leg through this liquid is pumped. It tries to move this magma in the upward direction. So this causes the circulation of the magma in the crystallizer body and more circulation then in an FC unit is achieved at the same power input.
- That is why all entire feed actually is circulated very efficiently and it is flowing in this direction.
- The fouling is less than that in the FC.
- The magma also flows out of the body through an annular zone between the skirt baffle and the wall of this crystallizer and enter such steam heated exchanger .
- The liquid flow path is shown by just arrow and recycles back to the crystallizer vessel. So that one part of magma is preheated.

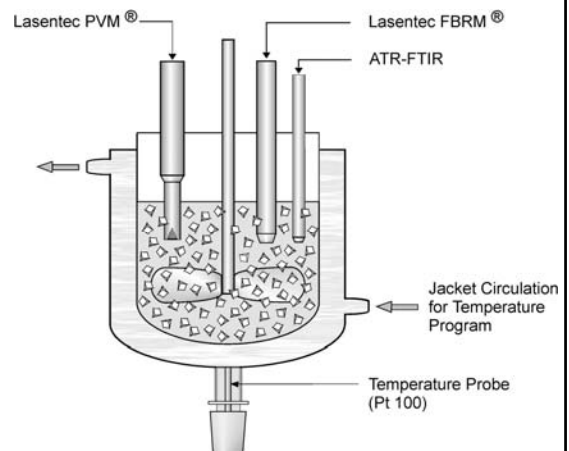


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Tank Crystallization

- Simple stirred batch reactor
- Advantages:
 - For pharmaceuticals, where uniform, well-defined crystals are important
 - High value, low volume products
- Disadvantage:
 - Labor is costly
 - Longer time
 - Nucleation and the size of crystals are difficult to control

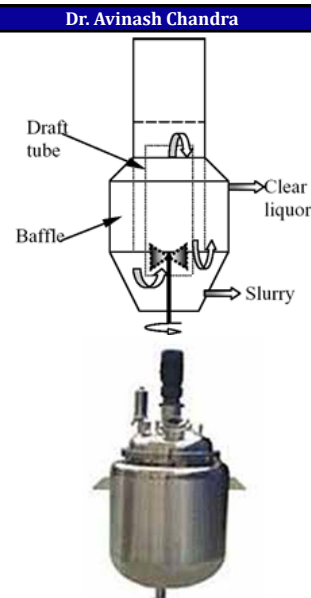


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- The next common crystallizer is the tank crystallizers.
- A tank crystallizer consists of a cylindrical tank provided with a cooling jacket/cooling coil.
- The cooling arrangement is provided only to generate the supersaturation.
- The hot feed is pumped into the tanks and the cooling liquor liquid is passed through the jacket or coil at a predetermined rate.
- The temperature differential between the liquor and cooling fluid should be low in order to reduce the deposition on the cooling surface.
- The use of an agitator keeps the crystal in the suspension and to prevent the excessive fouling of the heat transfer surface.
- The agitation is done throughout this cooling crystallization process.
- After a period of time, the mother liquor is drained and the crystals removed
- Has limited application; used to produce certain fine chemical and pharmaceutical products.



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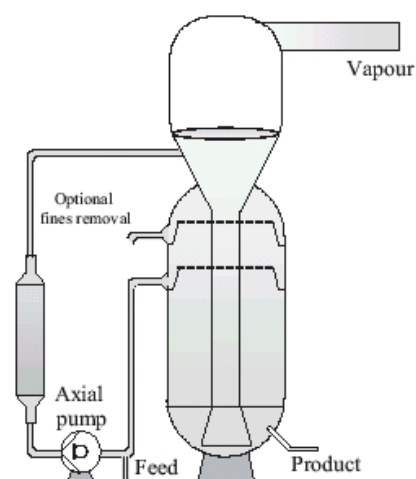
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Fluidized Bed**Advantages:**

- Large, uniform size

Disadvantages:

- Low production rate compared to Forced Circulation
 - velocity restricted by fluidized requirements
 - Supersaturation of liquid must be low
- Low birth rate of new crystals



<http://scholarsportal.info/pdflinks/04030101195012367.pdf>

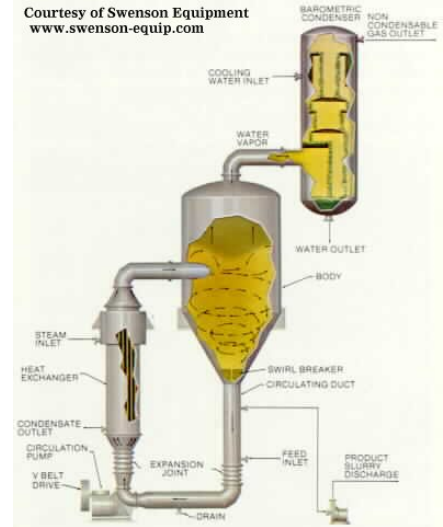
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Scraped surface crystallizers

- One type of scraped surface crystallizer is the Swenson-Walker crystallizer, which consists of an open trough 0.6 m wide with a semicircular bottom having a cooling jacket inside
- Slow-speed spiral agitator rotates and suspends the growing crystals on turning
- Blades pass close to the wall and break off any deposits of crystals on the cooled wall
- Used in crystallizing ice cream and plasticizing margarine



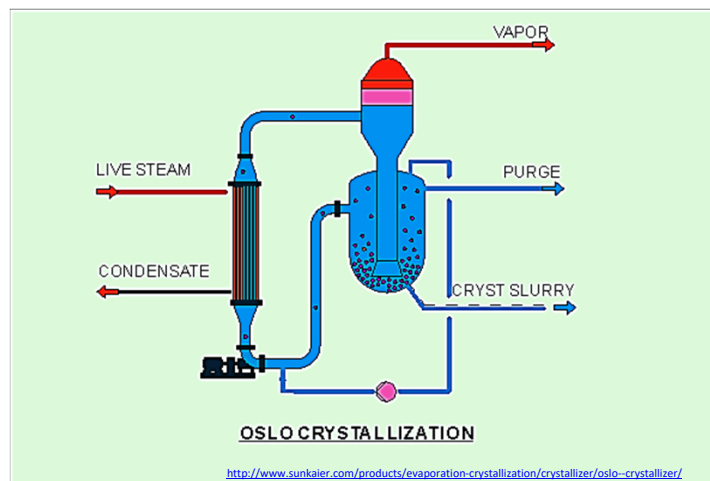
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Oslo crystallizer

- Supersaturation is generated by evaporation
- Circulating liquid is drawn by the screw pump down inside the tube side of condensing steam heater
- Heated liquid then flows into the vapor space, where flash evaporation occurs, giving some supersaturation

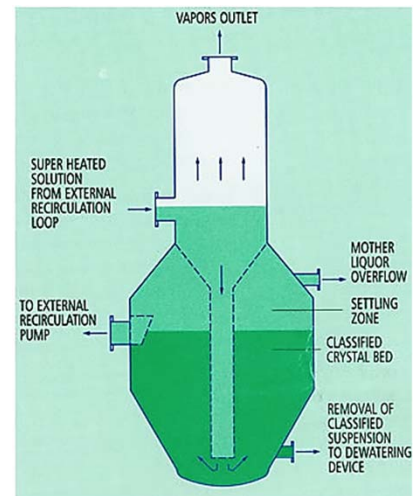


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- The supersaturated liquid flow down the downflow tube and then up through the bed fluidized and agitated crystals, which are growing in size
- The living saturated liquid then goes back as a recycle stream to the heater, where it is joined by the entering feed
- The larger crystals settle out and a slurry of crystals and mother liquor is withdrawn as product
- Also called **Circulating-Liquid Evaporated-Crystallizer**



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Choosing a Crystallizer

- Based on:
 - Properties of compound (solubility, temperature dependence)
 - Crystallization process
 - Required product specifications
- May also use:
 - Fines removal
 - Clear liquor
 - Product removal
 - Recycle loops

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Comparison of Crystallization and Precipitation

Description	Crystallization	Precipitation
Solubility	Wide range, usually medium to high	Sparingly soluble
Relative supersaturation	Low	High
Product morphology	Well-defined	Ill-defined
Product crystal size	Large	Small
Nucleation mechanism	Secondary	Primary
Nucleation rate	Low	High
Growth Rate	Wide Range	Low
Controllability	Controllable	Difficult to control

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References



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Mass Transfer

Theories for Mass Transfer Coefficients

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And Extraction - José Coca, Salvador Ordóñez and Eva Díaz

MASS TRANSFER OPERATIONS: ABSORPTION AND EXTRACTION

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- Lecture notes/ppt of Dr. Yahya Banat
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