

# Crystallization

Technoforce Plug Flow Crystallizer (TPFC)



## Concept

**Crystallization provides a process to produce solid crystalline compounds with high purity.**

### Crystal Size Distribution (CSD)

When crystals are formed out of a solution, impurities get concentrated in the mother liquor. The purity of the crystals after filtration or centrifugation depends upon how efficiently the mother liquor is washed from the filter cake. Uniformity in size or narrow crystal size distribution (CSD) helps in improving the washing and purity of crystals.

Certain important characteristics like flowability, Density and compressibility of the powders also depend upon CSD.

Conventional batch or continuous crystallizers have inherent limitations in achieving uniformity in crystal size. They tend to produce inconsistent results mainly due to uncontrolled nucleation.

### Primary nucleation

The lack of uniformity in mixing leads to unintended ingress beyond supersaturation line. It leads to uncontrolled primary nucleation and undesired amounts of fine crystals.

Even if the primary nucleation is avoided in the beginning with seeding, the risk remains in subsequent period due to lack of uniformity in saturation level.

### Secondary nucleation

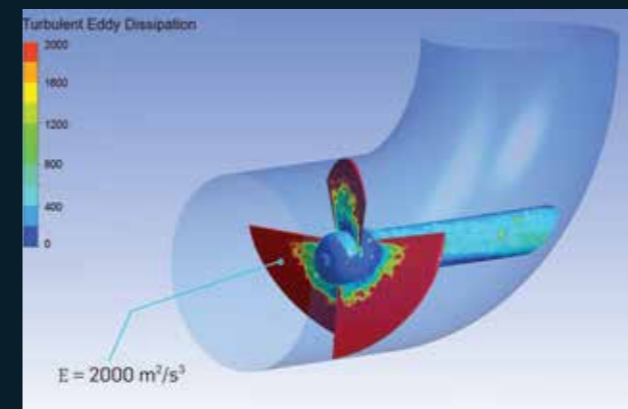
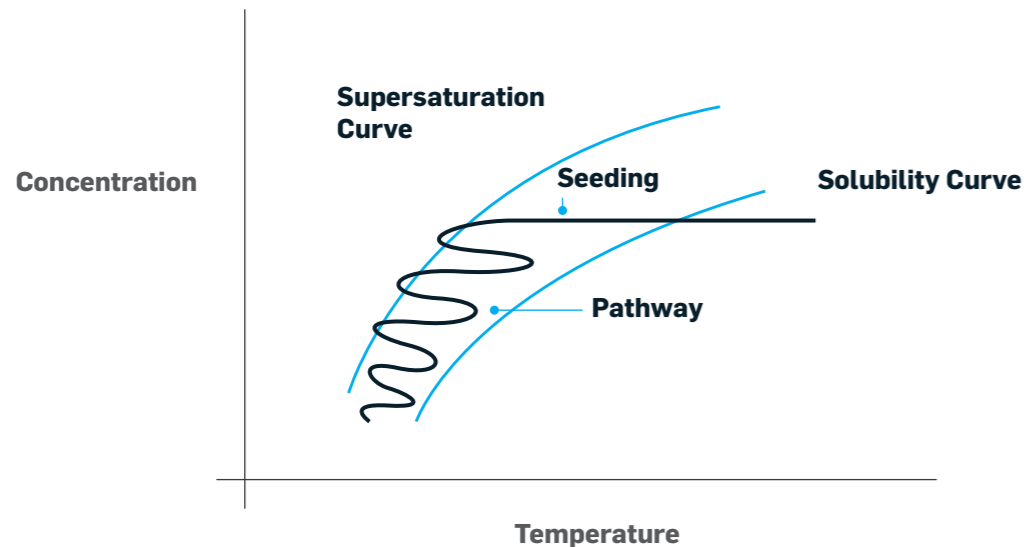
Improvement in uniformity in concentration is usually attempted by increasing power input to the agitator for better mixing or larger capacity pump for higher recirculation.

The conventional agitators and pump impellers dissipate large amounts of energy into the surrounding slurry.

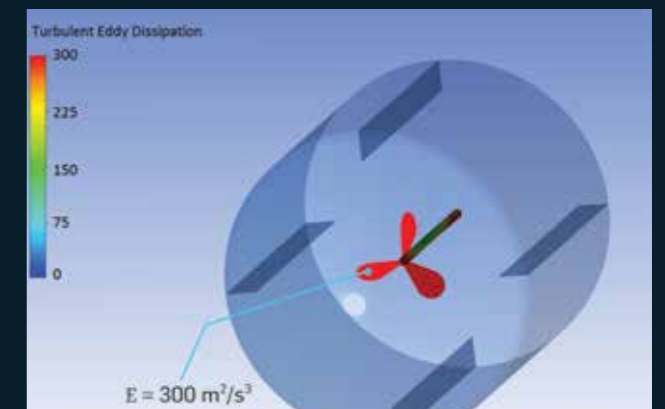
The large energy dissipation from impeller surfaces break the nearby crystals leading to secondary nucleation.

Thus the design of conventional crystallizer is a trade-off in power input. Higher power input minimises primary nucleation but increases secondary nucleation.

### Crystallization Pathway



Circulation Pump



Kettle

**An improved control of crystal size distribution is obtained in a continuous flow design that has negligible back-mixing with a residence time distribution approaching that of an ideal plug flow crystallizer.**

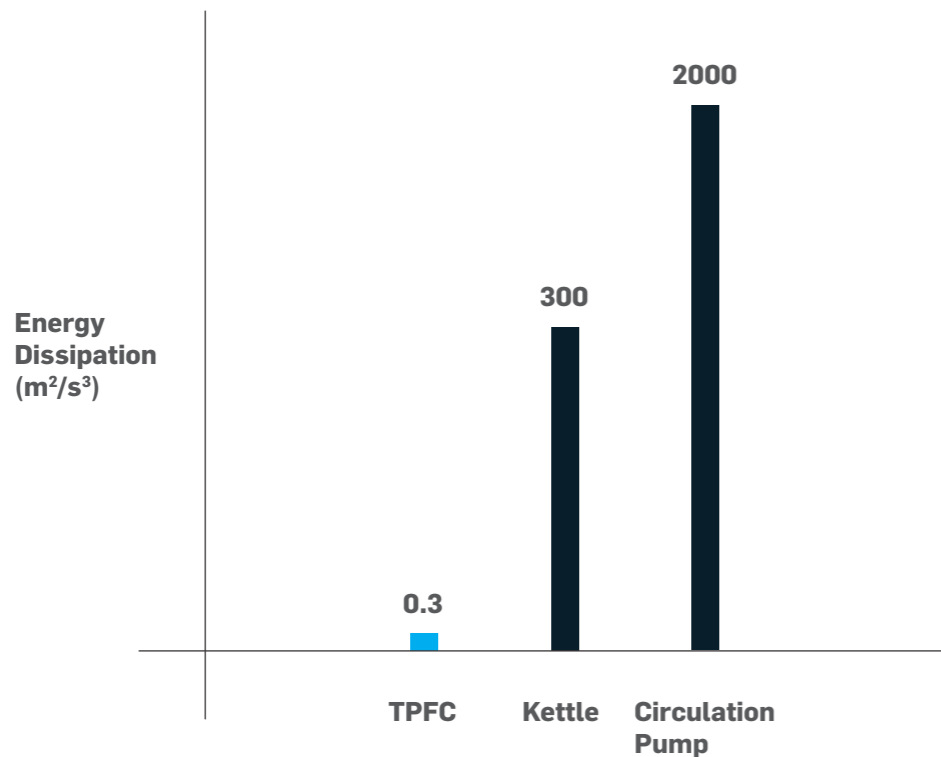
The trade off in design of conventional crystallizers is overcome by TPFC.

The unique agitator design of TPFC achieves uniformity in mixing with minimum power input. The uniform supersaturation eliminates uncontrolled primary nucleation.

The energy dissipation of agitator of TPFC is 1000 times less as compared to a batch kettle.

Therefore the formation of fine crystals due to breakage of existing crystals or secondary nucleation is minimised.

### Energy Dissipation



## Designed for improved results

**The novel continuous TPFC design has potential to provide improved control of crystal properties, improved process reproducibility and reduced scale-up risks.**

There are several compartmentalized stages in the TPFC. The feed flows from one stage to the next while experiencing different concentrations or temperatures. By the unique design of the rotor and the individual compartments, a plug flow with minimized back mixing is achieved.

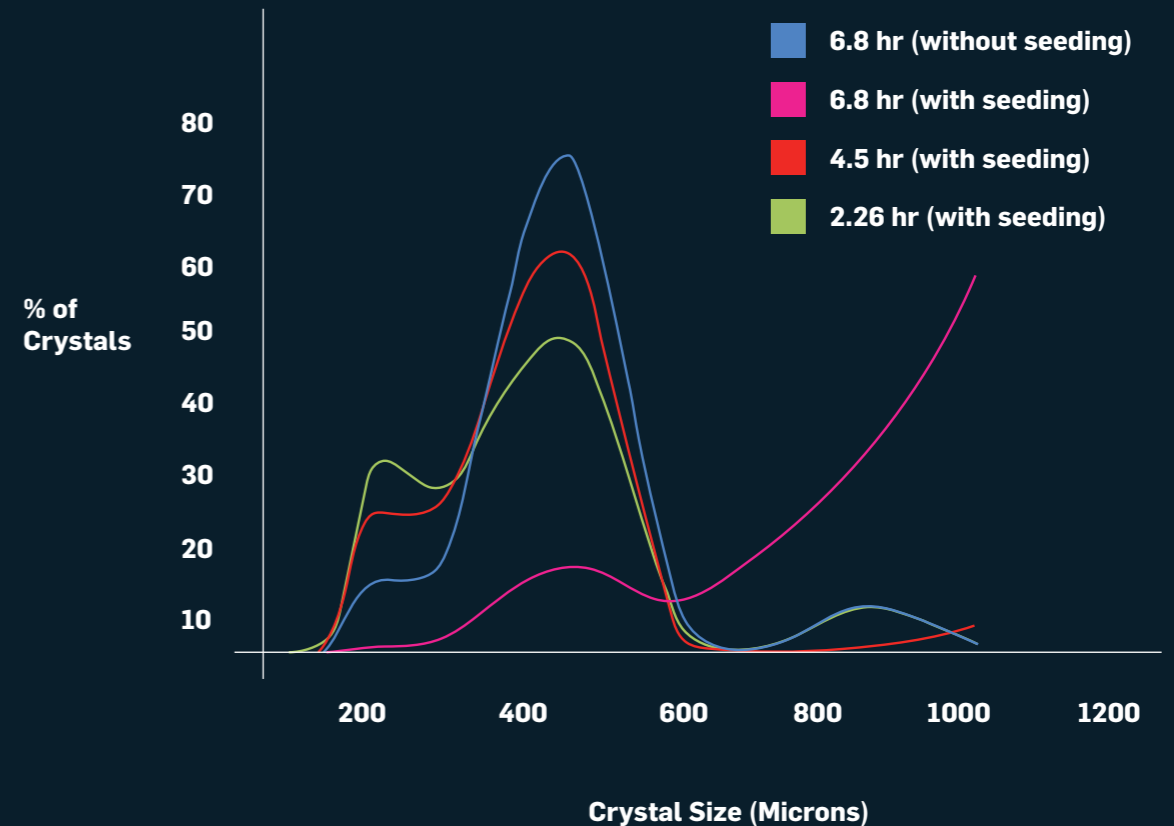
The plug flow characteristics ensure narrow residence time distribution and near equal time for the growth of individual crystals.

Crystal growth is controlled by varying the temperature profile along the length of the crystallizer.

The distinct capabilities of close control on saturation, reduced attrition and uniformity in residence time lead to significant improvements in CSD and final purity of the product.

With a judicious selection of residence time and seeding in a TPFC, it is now possible to get unparalleled control of CSD.

### Effect of Residence Time & Seeding





## Versatile Design

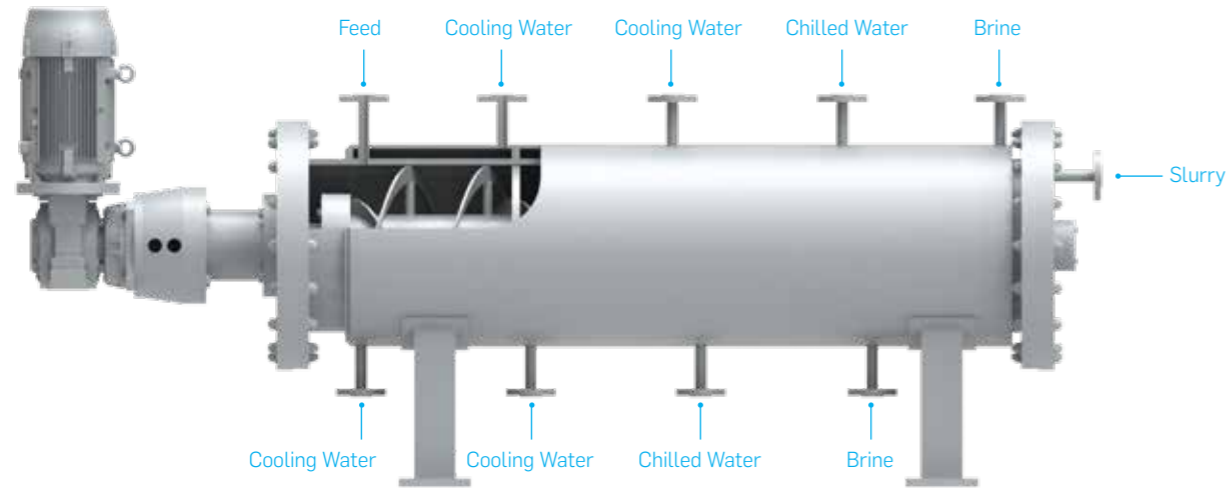
The TPFC is suitable for different configurations

When used as a cooling type crystallizer, each stage is provided with a separate jacket where the provided cooling media changes from normal water to chilled water and then to brine such that a controlled temperature profile is achieved. The utility costs are reduced by 50 % by resorting to this concept.

When used as an evaporative crystallizer the vapours are collectively taken out from all the stages.

As the feed passes through one stage to the next, the evaporation rate when matched with the crystal growth rate ensures avoidance of uncontrolled primary nucleation.

Similarly the TPFC can be easily adopted for anti-solvent crystallization.



## Validation tests and Scale-up

A systematic approach in understanding the saturation and supersaturation concentrations, crystal growth rates, crystal morphs in the laboratory scale investigations helps in planning the trials at the pilot plant scale.

Technoforce has the requisite laboratory scale and pilot plant scale crystallizers for conducting investigations.

With the analytical setups for CSD measurements, the operating parameters are optimised to get the desired powder characteristics and the same data is used for the scale-up.

The trials are backed by experienced technologists who are adept in the scale-up for production scale units.



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